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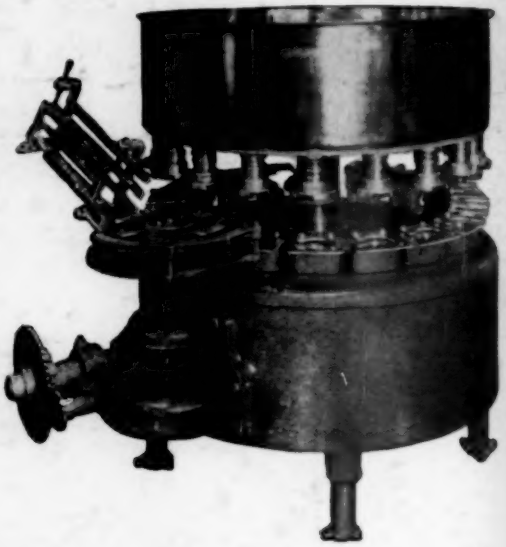

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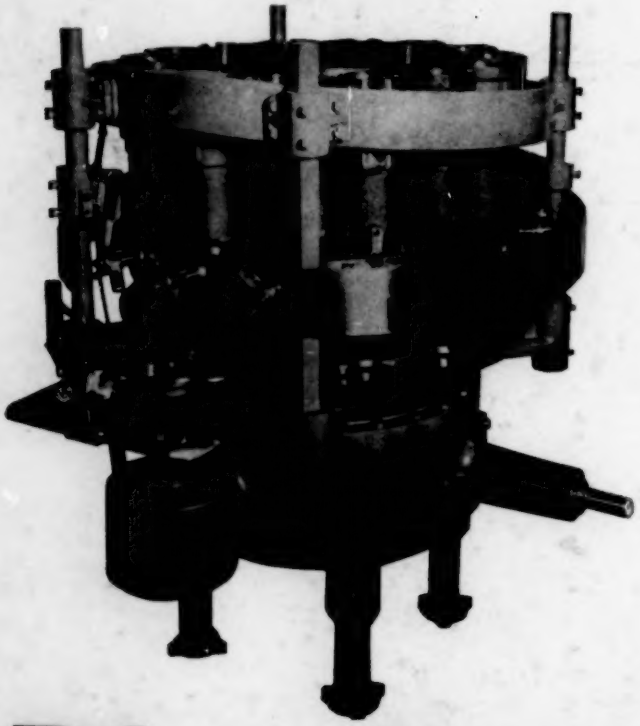

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Quality in Foods

Such words as truth, honor, hope and character are used in modern life without much thought being given to their meanings. In the field of food technology the words cleanliness, sanitation, dirt and quality are used and few would deny their desirability. The question may be raised as to how the good qualities indicated by them should be attained and how food control officials would know when they have attained them. To keep foods from becoming unnecessarily expensive nothing should be attempted in processing which increases cost without paying its way in an improved product.

It is not easy to separate frills and fancies from necessary efforts. If this is not done, the consumer pays for things which make no contribution to improvement in quality or safety. Furthermore, attention may be taken from important efforts and given to minor ones, which may only serve to confuse control officials and honest and progressive processors, many of whom have employed well trained, competent technologists. Processors and technologists have a right to ask why certain pronouncements are made and what they are supposed to accomplish. Some food control officials would profit by listening to food technologists in the processing plants from which come the products they are trying to "control." These technologists are just as competent and honest as are individuals in other professions.

Producers of certified milk believed for years that some of the requirements added materially to the cost of certified milk without making much contribution to improvement in quality. When it is remembered that pasteurization (the single process which makes milk safe) was scorned by those who defined certified milk the opinions of the producers were justified. In recent years *Brucella* infections and mastitis have forced producers to pasteurize milk. Health officials have long since recognized the need for making certain that it is properly and efficiently done because a vast difference exists between pasteurized milk and milk simply passed through a machine called a pasteurizer.

Another illustration of misguided effort is the program started about ten years ago in which the paper and paperboard industry, the products of which had never caused a single outbreak of disease, but which, on the other hand, had made striking contributions to decency and cleanliness in packaging and distribution of foods, were claimed to need microbiological control. This situation has been reviewed in other places but comment may be made about the proposal to establish a "plate count standard" for paper to be used in the food industry of not over 250 bacteria per gram of disintegrated board. No mention was made that these bacteria were only harmless aerobic spore formers, that paper during manufacture is subject to the most powerful germicidal processes and that presence of coliform bacteria has never been reported even in grades of paperboard made from badly contaminated raw materials. It is interesting that the two workers who proposed this count standard later stated that such counts on disintegrated paper and paperboard were inaccurate and without significance. Despite this reversal of opinion the standard has been accepted by those who prepared the standard milk ordinance.

How shall we attempt to measure "quality" of foods and how shall we know when it has been secured? For many years the "plate count" has been used in the dairy industry and is now being used for such foods as dried egg, dry milk, frozen vegetables, and ground beef. Much misconception exists about the meaning of "plate counts." It is wrong to allow this situation to grow and, in some cases, to be encouraged by food bacteriologists. The writer was once asked what plate count should be accepted as normal for a cured ham. Such a question is an absurdity. Recently his opinion was sought by a woman purchasing food for a children's home. A dealer whose milk showed a slightly lower plate count than the milk currently used approached her for the fluid milk account solely on the basis of the plate count of the last sampling. Such situations are difficult to explain to laymen when for many years the plate count has been played up as an index of quality where it is not. Food technologists know that a plate count is subject to so many errors that it is meaningless.

That plate counts are convenient whips to use by health officials and others on food producers and processors, is admitted. However, their use in this manner is basically dishonest in view of the fact that the counts are grossly inaccurate. When plate counts were used many years ago to control fluid milk they may have had some value because the "counts" were in the hundreds of thousands or even millions. Any reduction in numbers was helpful but today the situation is different with a count standard of 30,000 per ml. Several competent dairy technologists have wondered where this attempt to constantly lower the bacterial content of milk is going to end. One of them wondered whether zero numbers was the goal. Worship of numbers of bacteria without consideration of types leaves much to be desired.

The greatest difficulty with plate counts is that they are so inaccurate that they cannot be duplicated even by experienced workers. This was shown in a comprehensive investigation by G. S. Wilson in England, results of which were published in his *The Bacteriological Grading of Milk*. The various possibilities for error were emphasized. Wilson concluded that an allowance of ± 90 percent may have to be made because the experimental error is large. In view of this the plate count is no longer a statutory test and pasteurized milk must now meet the Methylene Blue Test and Phosphatase Test in which greater confidence is placed. The plate count is still used, however, in many municipalities in the United States.

Another element of quality which has much merit is presence of dirt and several types of foreign matter under the name of "filth." While these may not render a food deleterious to health, their presence indicates carelessness and bad housekeeping on the part of the processor or distributor. Activities of the Food and Drug Administration in recent years to eliminate filth are to be commended. While a clean food is not necessarily safe, it is in better position to be so than one which is consistently dirty. The dirt or sediment test is being looked upon with increasing favor by food technologists. No relation exists between the presence of dirt and to number and type of bacteria, although certain types of dirt and filth might be in better position to contribute special types of bacteria.

It is time in American food technology that first things be placed first and the maximum of good judgment and common sense be used in regulating foods. When are some bacteriologists and some food control officials going to wake up?

FRED W. TANNER
President I. F. T., 1945-'46

Destruction of Salmonella in Egg Albumen[a]

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The value of three methods (flash pasteurization, addition of hydrogen peroxide and storage at controlled temperature) are considered in this paper for the elimination of Salmonella from fermented egg albumen.

The commercial production of dried egg albumen has increased many fold within the past decade. During production the liquid egg white is subjected to a fermentation process in order to promote stability in the dried state. As this process requires incubation at room temperature for two to seven days, ample time is provided to permit enormous changes in bacterial populations. Organisms of the genera *Aerobacter* and *Escherichia* comprise the predominant flora but other bacteria introduced with the egg, from handlers, or supplied by the fermentation contribute to the total load of organisms present at the time of drying.

Dried albumen has a number of applications such as in nougat creams, marshmallow whip, icings, and in other confections. In these the product is uncooked or insufficiently cooked to kill pathogenic bacteria. Recently many investigators have reported the occurrence of *Salmonella* in egg products. These two factors have served to stimulate workers to search for methods for eliminating these undesirable microorganisms from the product.

Of the various methods of producing products of low bacterial count that have been applied to egg products, only a few have been reported for treating albumen. Perhaps the most desirable is the pasteurization process described by Winter, et al *(10)*. Heating albumen at 134° F. (56.7° C.) for 1.6 minutes destroyed all coliform bacteria according to Wilkin and Winter *(8)*. However, these workers reported that pasteurization adversely affected beating and properties although no data were presented to substantiate this view. The work, as reported, was based on laboratory scale experiments.

Slosberg, et al *(6)* found that time and temperature were factors in heating albumen if loss of desired functional properties is to be avoided. Permissible times and temperatures were determined for egg white by performance tests using the meringue beating rate and angel cake volume as criteria.

According to a report released by the Office of Technical Services *(2)* hydrogen peroxide is being used in Italy to sterilize milk. Conquest *(4)* suggested that liquid egg might also be sterilized by H_2O_2. The residual hydrogen peroxide from the treatment can be removed by catalase.

Gibbons and Moore *(5)* report that over 99% of organisms of the genus *Salmonella* in liquid whole egg are killed during drying. Further, they found that viable organisms survived for longer periods of time at lower temperatures of storage. In the course of a three month period experimentally infected whole egg showed a 75% reduction at 20° F. (—1.1° C.); and 97% at 45° F. (7.2° C.). Wilson *(9)* recently has indicated that *Salmonella* remains viable in whole egg powder for long periods of time at temperatures necessary to maintain dried egg quality and that the survival period decreases, as does solubility and quality of the egg, with increase in temperature of storage.

The present study was undertaken with the view toward development of a method that would permit the production of a product free of *Salmonella* while, at the same time, retaining desirable functional properties.

Materials and Methods

The egg albumen used in the present tests consisted of fresh whites or sugar-free whites from laboratory- or commercial-scale fermentations. In the latter case, after the albumen had been fermented, it was adjusted to pH 9.0 with dilute ammonium hydroxide to prevent further rapid growth of organisms responsible for the fermentation.

Bacteriological Methods. Bacterial analyses for total and for coliform counts were made at the plant and again at the experiment station bacteriological laboratory. Nutrient agar and desoxycholate citrate agar were employed for the standard and coliform counts respectively. Eosin methylene blue (E.M.B.) agar was used at the laboratory. Both coliform and total counts were obtained on this medium. The liquid and dried albumen were examined for *Salmonella* using Selenite-F enrichment media, SS agar and Krumwiede's Triple Sugar agar. A microscopic examination was made of typical colonies. The urease test was employed for questionable reactors. Quantitative data for numbers of *Salmonella* were obtained through use of a "most probable numbers" technique described by Ayres *(3)*. *Salmonella* studies were made only at the station laboratory.

Coliform counts were obtained by direct plating in EMB agar; presence of *Salmonella* was determined after enrichment in Selenite-F and subculture to SS agar. For the albumen fermented with yeast, malt agar plates were used as a subculture medium.

Performance Tests. Initially, high temperature storage experiments were made on dried albumen in sealed glass jars and the product observed from time to time for changes in functional properties. Beating rates and changes in solubility were used as criteria of quality.

The whipping test used to determine the effect of the various treatments on the dried albumen was that normally used by the trade as one means of measuring albumen quality. One and one-half ounces of dried flake

[a] Journal Paper No. J. 1601 of the Iowa Agricultural Experiment Station, Ames, Iowa. Project No. 970.

albumen were reconstituted in 15 ounces of water at 70° F. (21.1° C.) for three to six hours. The solubility of the albumen was observed carefully at this time inasmuch as the rate at which the product goes into solution and the amount of undissolved coagulum remaining prior to whipping are important criteria of quality.

The reconstituted liquid was whipped in a ten quart Hobart mixer for ninety seconds at second speed and ninety seconds at high speed. The depth of the foam and the texture of the foam were measured.

Pasteurization Tests. Laboratory scale pasteurizations were run according to the technique outlined by Winter, et al *(10)*. In the commercial pasteurization trials, a 500 lb. stainless steel vat was filled with liquid albumen and permitted to ferment spontaneously. When sugar-free, the pH was adjusted and samples were "flash" heated in a commercial plate-type heat exchanger to 135°, 137.5°, 139°, 140°, and 142° F. (57°, 58.6°, 59.5°, 60°, and 61.1° C.) respectively, or heated and held for one minute or for ten minutes. Immediately after pasteurization, the liquid was cooled to 90° F. (32.2° C.) and poured on aluminum trays then dried at approximately 120-125° F. (48.8-51.7° C.).

Samples were collected from the commercial runs immediately before and after pasteurization. For the most part samples were dried and the dry product reconstituted for test. However, some liquid samples were frozen. These were placed in a carton containing dry ice and shipped to the laboratory by air express. Four ounce screw cap glass containers were used for shipment of dry and frozen albumen samples.

Hydrogen peroxide and catalase. Tests were run for 15 and for 30 minutes using 0.05, 0.1 and 0.2% hydrogen peroxide in 50 ml. of fresh or fermented albumen to which one drop of a 20 hour broth suspension of *Salmonella* sp. had been added. Residual H_2O_2 was then removed by means of an excess of catalase.

Catalase activity was determined by the method advocated by the Chemical Research and Development Dept., Armour and Co. *(1)*. Technical catalase could not be used as supplied due to its high load of microorganisms. However, by adjusting the catalase to pH 11.2 and passing through a Seitz filter the bacterial contamination could be removed; much of the catalase activity was thereby retained *(7)*.

The evolution of oxygen from the H_2O_2 treatment caused considerable foam. The amount was measured by placing 50 ml. of the material tested in a 100 ml. graduate.

Storage Tests. Dried fermented albumen known to be highly contaminated was stored at room temperature and at several higher temperatures. After storage, total coliform and *Salmonella* counts were determined.

Later, a similar experiment was made using barrels of dried fermented albumen stored at a temperature of 120° F. (48.9° C.) for varying periods of time.

Results

Pasteurization. Table 1 shows that flash pasteurization eliminated *Salmonella* from fermented liquid albumen when temperatures higher than 137.5° F. (58.6° C.) were employed.

TABLE 1

Effect of Pasteurization Time and Temperature on Destruction of Bacteria in Fermented Albumen

Temperature	Holding Time	Liquid Albumen		Dried Product After Reconstitution at the Laboratory	
		Total Count	Coliform [b]	Total Count	Presence of Salmonella
Control	---	204,000,000	1,000	1,800,000	+
135° F.	1 min.	16,000	0	200	+
135° F.	10 min.	3,000	0	134,000	+
137.5° F.	flash	320,000	59,000	12,000	+
139° F.	flash	1,100,000	125,000	100	—
140° F.	flash	1,070,000	0	0	—
142° F.	flash	3,000,000	0	8,800	—

[b] Plated on Desoxycholate agar at the plant.

On the other hand, reconstituted fermented albumen was not found to be free of *Salmonella* even after the product had been held at a pasteurization temperature of 135° F. (57° C.) for ten minutes. Also the low counts in the liquid albumen contrasted sharply with similar analyses made of the dry product. The increase in count in the finished dry product in the lot pasteurized for ten minutes at 135° F. raised the suspicion that this albumen was recontaminated after pasteurization. Examination of containers, pumps, and sanitary lines used to get the liquids to the drying trays revealed presence of coliform organisms, confirming the suspicion. In addition, an inspection of storage vessels and drying pans indicated that some of these utensils harbored large numbers of microorganisms. In another study, a permanent flora was found to persist in the wax layer commonly used on drying pans to prevent sticking.

As a consequence of these difficulties, further large scale pasteurization trials were not made.

Hydrogen Peroxide. Addition of hydrogen peroxide to fermented liquid egg white and removal of the residual hydrogen peroxide with excess catalase resulted in marked reduction in bacterial population (Table 2).

TABLE 2

Effect of Hydrogen Peroxide on Bacterial Count of Egg Albumen

Sample Tested	Conc'n H_2O_2	Test time min.	Total count on E. M. B. agar	Presence of Salmonella	Foam ml.
Fermented albumen	0.0%	0	320,000,000	+	
	0.05%	15	17,000,000	+	trace
		30	4,500,000	+	2
	0.1%	15	1,100	—	8
		30	0	—	11
	0.2%	15	0	—	27
		30	0	—	30
Fermented albumen (yeast)	0.0%	0	810,000	+	
	0.05%	15	32,000	+	3
		30	48,000	+	5
	0.1%	15	0	—	18
		30	0	—	20
	0.2%	15	0	—	42
		30	0	—	45

As little as 0.1% hydrogen peroxide for fifteen minutes eliminated all viable organisms of the *Salmonella* group. With 0.1% H_2O_2 for 30 minutes or with 0.2% H_2O_2 for 15 or 30 minutes, foam was considered excessive. When catalase was added to decompose the remaining hydrogen peroxide, still more foam was evolved.

Storage Tests. A number of the samples stored at room temperature were known to have high coliform counts immediately after drying. These were tested after varying periods of time to determine whether or not coliform organisms and also *Salmonella* could be destroyed thereby. Table 3 shows the decrease in counts at the end of different storage periods with several different samples.

The results in Table 3 indicate that storage in the dried state at ordinary room temperatures cannot be relied upon to free the albumen from *Salmonella* in the fermented product. Considerable numbers of *Salmonella* persisted in samples kept for eight months in the dried state. Even in dried albumen stored for 18 mos. a few potential enteric pathogens were present. Of some interest are the comparative numbers of organisms of the *Salmonella* group in egg white broken out during the early spring months, late spring, and during the summer. Of equal interest are the large numbers of *Salmonella* that continue to survive for long periods in the frozen state.

TABLE 3

Survival of Salmonella in Dried Albumen Stored at Room Temperature (65° F. to 90° F.)

History of Sample	Time of Storage in Dried State	Counts after Storage g.[a]
Fresh egg white; December	1 mo.	0
Spring egg white frozen for 8 mos.	1½ mos.	6 780
Summer egg white frozen for 3 mos.	4 mos.	100,000 50,000
Fresh egg white; June	8 mos.	.25 .25
Fresh egg white; Spring	8 mos.	0
Fresh egg white; Spring	15 mos.	.037
Fresh egg white; Spring	18 mos.	.018

[a] All lots known to have *Salmonella* at time of storage in dried state. Since previous holding conditions varied, initial *Salmonella* counts were not the same.

Increased temperatures were employed to test their effect upon the enteric pathogens that may have been present in the several lots of albumen examined. These data are given in Table 4. Storage of albumen known to contain *Salmonella* for a period of eight days at 130° F. was effective in eliminating viable organisms of the *Salmonella* group. No survivors were found in albumen stored at 120° F. for twenty days or in dried fermented egg white after four days' storage at 135° F. Also, the number of coliforms was greatly reduced in all samples stored at these higher temperatures.

The functional properties deteriorated slowly at room temperature. The changes in solubility and beating rate were increased directly by storage temperature. The extent of the change varied with various lots of albumen, but heated samples normally were observed to be less soluble than controls. Certain lots showed insolubility more quickly than did others; if the albumen originally showed little or no coagulated flake, the product remained soluble for longer periods. As would be expected, those lots having the highest moisture content became insoluble when subjected to heat more readily than did similar samples containing lower percentages of moisture. It was observed that the beating rate generally did not decrease within three weeks when the temperature was held at 120° F. At 130° F., satisfactory beating rate was retained for eight days while at 135° F. the rate began to decrease after only four days. Similar results were observed regarding solubility. At 130° F. and 135° F. a decrease in rate of reconstitution was observed after eight and four days respectively.

Discussion

Of the three methods studied for the purpose of removing *Salmonella* from fermented egg albumen, two were concerned with destruction of the potential pathogens in the fermented liquid albumen prior to drying. The other simply involved storage of the dried albumen for controlled periods of time.

A commercial scale pasteurization method is described which resulted in freeing the fermented liquid albumen from *Salmonella* without seriously impairing certain of its functional properties. Insofar as solubility and whipping properties were concerned, a flash pasteurization at 139° F. was found satisfactory for destroying enteric pathogens.

There were several instances wherein the sample again became contaminated by a heterogenous flora after pasteurization. The source of this contamination was traced to vessels used to transfer or hold the albumen before drying and to the drying trays.

TABLE 4

Survival of Salmonella and Total Organisms in Dried Albumen Stored at Elevated Temperatures

Temp. of storage	Sample	No. of Salmonella/g. dry albumen							Total count/g. dry albumen[d]											
									(Station Laboratory)						(Plant)					
		Time in days							Time in days						Time in days					
		0	2	4	6	8	10	20	0	2	4	8	10	14	0	1	2	4	8	14
120° F	Flake alb.	1900	....	....	....	....	....	0												
	Flake alb.	1750	....	....	....	41[e]	0[e]	0							12,300,000	....	....	....	....	40,000
130° F.	Flake alb.	+	....	....	....	0			3,460,000	....	....	320,000			10,800,000	....	650,000	490,000	100,000	
	Flake alb.	+	....	....	....	0			3,460,000	....	....	130,000	....	7,000	3,070,000	....	730,000	51,000	300	
	Flake alb.	250	....	....	....	....	0		4,700	....	....	....	500		70,000	....	500	1,000	0	
	Gran. alb.	700	....	....	....	....	0		1,800	....	....	....	0							
	Flake alb.	6700	60	25	9	0	0		143,000	240	0	0	0							
135° F.	Flake alb.	3000	....	0					880,000	....	0				7,400,000	360,000	100,000	1,000		
	Gran. alb.	2250	....	0					297,000	....	0				580,000	212,000	78,000	5,000		

[d] In every case where coliform counts were made, they were negative after 48 hrs. at the elevated temp. Initial counts were as high as 56,000.
[e] Tests made after storage at 120° F. of 7 and 14 days respectively.

Hydrogen peroxide and catalase show some promise in eliminating all microbiological contamination from liquid albumen. In this connection, foaming is a serious problem. It may be possible to use a small amount of hydrogen peroxide for a longer period of exposure and so cut down on the foam produced.

While the effect of hydrogen peroxide is recognized to be transitory, such stop-gap removal of a focus of *Salmonella* would serve a useful purpose in ridding the product of possible pathogenic contaminants in the liquid egg white. After drying, the albumen no longer would be subject to gross contamination by non-spore-forming microorganisms.

It is common knowledge that bacterial survival is inversely proportional to the temperature of storage. Gibbons and Moore *(5)* employed room temperature and lower temperatures with dried whole egg powders. They observed reduction within short periods of time. However, fermented dried albumen has a much more populous flora, and if the counts shown in this study are typical, a greater number of *Salmonella* per gram of product. With this in mind, one would expect a greater number of survivors in a comparable period of time. Also, much of the fermented dried albumen is in the form of flakes and not powder as is the case with spray dried whole egg powder. The protective action of such agglomerates would prolong periods of survival. Then, too, the temperature of drying albumen must be kept lower than is necessary with whole eggs and so fewer bacteria would be killed during the drying action.

In one experimentally infected sample of dried whole egg powder Gibbons and Moore *(5)* found that after storage at 37° C. for three days no organisms could be detected in ten gram samples in three days or in twenty gram samples in five days although they had included *S. bareilly* at a level of thirty organisms per gram.

By applying temperatures higher than room temperature for short periods of time, results of present experiments indicate that it may be possible to eliminate *Salmonella* from dried fermented albumen without seriously impairing functional properties of the albumen. At a temperature of 130° F. for eight days, all organisms of the *Salmonella* group were destroyed without noticeable decrease in beating properties in the heated product. Reconstitution of the dried albumen was still possible.

Summary

Three methods were studied that would free fermented albumen of organisms of the *Salmonella* group.

Flash pasteurization at 139° F. on a commercial scale was successful in destroying viable *Salmonella*.

Some evidence is indicated that liquid albumen is recontaminated in holding vessels and in drying trays.

Hydrogen peroxide at levels of 0.1 and 0.2% followed by removal of the residual H_2O_2 with excess catalase destroyed all organisms of the *Salmonella* group but produced excessive amounts of foam under the test conditions.

Storage of dried fermented albumen at room temperatures was not successful in ridding the product of living *Salmonella*. Storage at 120°, 130° and 135° F. for 20, 8 and 4 days, respectively, freed the products of potential enteric pathogens. Beating rates were retained in each of the products during the same time intervals. Solubility properties were still satisfactory after viable *Salmonella* had been eliminated.

Elevated temperature storage of the dried fermented albumen eliminates the danger of recontamination during the drying process encountered when liquid albumen is sterilized.

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B-Complex Vitamins in Meat

II. The Influence of Different Methods of Thawing Frozen Steaks Upon Their Palatability and Vitamin Content[a]

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Frozen steaks were thawed in a refrigerator at 73° C., at room temperature, and in running tap water. In general the results did not differ, with the exception of thawing in water, which resulted in significant losses in both palatability and vitamin content.

Most meats are good sources of the B vitamins. Vail and Westerman *(9)* and other investigators have shown that the different cooking processes affect the retention of the vitamins in the meat. The preservation of meat by frozen storage has become a common practice and may have some influence on the vitamin retention. Several different methods of thawing the meat after removal from frozen storage have been used. It is often thawed in the refrigerator, in the warming oven, at room temperature, in water or by other means. The temperature and length of time of thawing, as well as the washing out of certain constituents from the surface may have an effect upon the palatability and nutritive value of the meat after it is cooked. Few data were available on this subject; therefore it was deemed advisable to determine the palatability and retention of some of the B vitamins in steaks which had been stored in the frozen condition and thawed by different methods. Data on four methods of thawing frozen steaks (i.e., in the refrigerator, at room temperature, in the warming oven at 73° C. and in running tap water) are reported in this paper.

Experimental Procedure

The rounds from three beef animals were obtained from the packing house. Nine steaks were cut from each round. They were given numbers 1 to 9 and each pair labeled right and left. Steaks numbered 2, 5 and 8 were used for the raw analyses, while the others were wrapped and placed in frozen storage for a short time. The bones and fat were removed from the raw steaks and the entire lean portion was ground 3 times in a food chopper; it was thoroughly mixed and samples were taken for analyses. Before cooking, the frozen steaks were removed from storage and thawed in the refrigerator, at room temperature, at 73° C., or in running tap water. In order that the data might be analyzed statistically, a random sampling design for thawing the steaks was made and is shown in Table 1. The arrangement follows a symmetrical pairs design described by Yates *(10)*.

The steaks were approximately of the same thickness. After thawing they were browned, 60 ml. of water was added, and they were braised in a covered roaster for 60 minutes in an oven at 325° F. Then the cooked steaks were removed from the drippings and weighed. A small portion of the same muscle was judged by a committee of 5 individuals on the desirability and intensity of the flavor and aroma of the fat and lean, as well as the juiciness and tenderness of the meat. The bone and fat were removed from the other portion and the lean was ground and mixed in the same manner as the raw samples. The drippings were cooled, the fat removed and the remainder saved for vitamin analyses.

The meat and drippings were assayed for 4 of the B vitamins. The thiochrome method of Hennessy *(3)* was used for the thiamine determinations. Riboflavin was determined by the fluorometric method of Peterson, Brady and Shaw *(6)*. The microbiological methods as described by Strong, Feeney and Earle *(8)* and Krehl, Strong and Elvehjem *(5)* were used to determine pantothenic and nicotinic acids respectively. Fat was determined according to the AOAC method *(1)* and moisture by the method recommended for the Cooperative Meat Investigations *(7)*. Fat and moisture determinations were made in order to report the vitamin content of the meat on the dry and fat-free basis.

Results and Discussion

The results of the thawing and cooking processes as well as the palatability tests are shown in Table 2. There was considerable difference in the average time required to thaw the steaks by the four different methods. Steaks thawed in the refrigerator required on the average 10.48 hours per pound to thaw. Those thawed in running tap water required only 0.35 hour per pound. However, the average percentage thawing loss was least in the steaks thawed at 73° C., while those thawed in water absorbed water and gained in weight. There were also some differences in the average cooking losses. The greatest cooking loss was found in the

TABLE 1

Random Sampling Design for Thawing Steaks

		1	2	3	4	5	6	7	8	9
Animal No. 1	L	b	o	b	a	o	c	c	o	a
	R	c	o	d	b	o	a	d	o	d
Animal No. 2	L	b	o	c	d	o	a	b	o	c
	R	d	o	b	c	o	d	a	o	a
Animal No. 3	L	c	o	d	a	o	b	d	o	b
	R	a	o	b	d	o	a	c	o	c

a = thawed at 73 degrees C.
b = thawed at rom temperature.
c = thawed in refrigerator.
d = thawed in water.
o = the cuts used for raw analysis.

[a] Contribution No. 145 of the Department of Home Economics. Supported by a grant from The Refrigeration Research Foundation (Inc.) and by Purnell Funds.

TABLE 2

Data on Thawing and Cooking
Average figures on steaks from 3 animals.

	Methods of Thawing			
	Refrigerator Temperature	Room Temperature	Oven 73° C.	Water
Thawing time, hr./lb.	10.48	4.03	0.80	0.35
Percentage thawing loss	0.65	1.42	0.55	+1.44*
Percentage cooking loss	32.50	34.10	34.30	39.70
Palatability Tests	Maximum Score 7			
Desirability				
Flavor of lean	6.1	5.9	5.9	5.3
Flavor of fat	5.5	5.5	5.8	4.8†
Aroma	6.2	6.0	6.0	5.5
Intensity				
Flavor of lean	5.5	5.3	5.5	4.9
Flavor of fat	5.5	5.2	5.2	4.8
Aroma	5.5	5.4	5.7	5.2
Juiciness				
Quantity	5.2	5.2	4.9	4.3†
Quality	4.9	5.0	5.0	4.3†
Tenderness	5.0	5.0	5.0	5.0

* Percentage thawing gain.
† F test showed significant differences.

steaks thawed in water and the least cooking loss in those thawed in the refrigerator. A statistical analysis of the palatability data was made. The F test was applied, followed by Duncan's *(2)* procedure for ranked variates. These tests indicated that the desirability of the flavor of the fat and the quality and quantity of juiciness of the steaks thawed in water were significantly lower than the same qualities in steaks thawed by the other methods. The interaction between animals and treatments was so large that none of the thawing methods showed consistently better results for the other palatability tests.

Data on the vitamin content of the raw steaks and the meat after thawing and braising are given in Table 3. Here the statistical plan shown in Table 1 is used to compare the content of vitamins in raw steaks with that in steaks thawed by the different methods. The results of the analyses of the meat from 3 animals are given. An analysis of variance was made on the data to determine whether or not the various methods of thawing affected the vitamin content of the meat. As shown in Table 3 the F values for thiamine, riboflavin and nicotinic acid showed no significant differences in the different methods of thawing. There were significant differences in the pantothenic acid content of the steaks thawed by some of the different methods. Thawing in the refrigerator and at room temperature produced no significant difference in the pantothenic acid retention. Thawing in the refrigerator led to significantly more retention than thawing at 73° C. ($P<.05$) and thawing in running water ($P<.01$). Thawing at room temperature gave significantly more retention than thawing at 73° C. ($P<.05$) and thawing in running water ($P<.01$). The methods of thawing in the refrigerator and at room temperature were best and perhaps in that order. Thawing at 73° C. and in running water were poorest, perhaps in that order. The observed differences between thawing in the refrigerator and at room temperature and between thawing at 73° C. and in running water were not statistically significant.

The percentage retention of the vitamins in the steaks after thawing and braising is shown in Table 3. It may be noted that there is less percentage retention of thiamine, pantothenic and nicotinic acids in the steaks thawed in running water than in those thawed by the other methods. Those thawed at 73° C. showed less

TABLE 3

Vitamin Content of Raw and Thawed Braised Steaks
Comparisons made according to statistical plan in Table 1.
Data reported on a dry, fat-free basis.

Animal No 1		Thiamine		Riboflavin		Pantothenic Acid		Nicotinic Acid	
		μg/g	% retained	μg/g	% retained	μg/g	% retained	μg/g	% retained
Raw	oR2	7.32		3.02		12.15		132.79	
Refrig.	cR1	3.11	45.21	6.12	203.6	9.62	79.18	123.73	93.17
Water	dR3	2.66	36.34	6.31	208.9	8.94	73.58	83.85	63.12
Raw	oL2	6.69		2.51		11.17		136.42	
Room	bL1	2.88	43.05	5.14	204.78	9.54	85.41	115.38	84.57
Room	bL3	3.22	48.13	5.11	203.58	9.48	84.87	116.75	85.58
Raw	oR5	4.50		2.80		12.68		133.27	
73° C.	aR6	3.70	82.22	4.78	170.8	7.37	58.12	86.63	65.00
Room	bR4	2.81	62.44	4.83	172.5	9.88	77.92	101.25	75.97
Raw	oL5	7.13		2.42		12.14		223.60	
73° C.	aL4	2.26	31.69	5.49	226.8	8.71	71.75	93.05	41.61
Refrig.	cL6	3.02	42.36	5.88	242.9	10.87	89.53	103.24	46.17
Raw	oR8	8.02		2.48		11.67		192.03	
Water	dR7	2.54	31.67	5.95	239.9	9.36	80.2	83.97	43.73
Water	dR9	2.30	28.67	5.74	231.5	8.32	71.29	88.82	46.25
Raw	oL8	7.29		2.36		11.50		176.40	
73° C.	aL9	3.20	43.89	6.48	274.5	8.15	70.87	87.97	49.87
Refrig.	cL7	5.03	68.99	6.39	270.7	10.21	88.78	163.68	92.78
Animal No. 2									
Raw	oR2	4.11		5.82		11.32		163.72	
Water	dR1	2.96	72.02	5.86	100.69	7.60	67.14	126.72	77.40
Room	bR3	3.24	78.83	6.26	107.56	10.52	92.93	107.39	65.59
Raw	oL2	3.15		6.87		10.93		179.09	
Room	bL1	2.98	94.60	6.08	88.50	8.76	80.15	135.09	75.43
Refrig.	cL3	3.92	124.44	5.75	83.70	11.87	108.60	140.63	78.52
Raw	oR5	4.84		7.62		12.68		164.99	
Refrig.	cR4	3.44	71.07	5.81	76.25	11.08	87.38	134.08	81.27
Water	dR6	2.67	55.17	5.75	75.46	7.80	61.51	117.87	71.44
Raw	oL5	3.88		5.28		10.30		175.90	
Water	dL4	3.15	81.19	5.72	108.33	7.49	72.72	127.79	72.65
73° C.	aL6	3.15	81.19	5.72	108.33	9.74	94.56	121.31	68.97
Raw	oR8	4.25		5.19		13.99		154.83	
73° C.	aR7	2.76	64.94	5.63	108.48	8.82	63.05	133.10	85.97
73° C.	aR9	2.72	64.00	5.67	109.25	8.60	61.47	119.92	77.45
Raw	oL8	3.72		5.67		14.61		158.61	
Room	bL7	3.17	85.22	5.79	102.12	10.58	72.42	90.53	57.08
Refrig.	cL9	2.98	80.11	5.22	92.06	9.46	64.75	123.76	78.03
Animal No. 3									
Raw	oR2	5.83		5.09		18.41		177.07	
73° C.	aR1	2.61	44.77	4.47	87.82	8.08	43.89	117.53	66.37
Room	bR3	3.02	51.80	4.32	84.87	11.86	64.42	119.45	67.46
Raw	oL2	6.49		6.40		20.48		165.12	
Refrig.	cL1	2.28	35.13	5.50	85.94	10.37	50.63	100.94	61.13
Water	dL3	3.03	46.69	4.42	69.06	7.68	37.50	104.87	63.51
Raw	oR5	6.93		5.33		17.37		172.33	
Water	dR4	2.18	31.46	3.99	74.86	8.43	48.53	116.76	67.75
73° C.	aR6	2.55	36.80	4.24	79.55	10.16	58.49	121.17	70.31
Raw	oL5	4.53		4.99		15.61		145.45	
73° C.	aL4	2.77	61.15	4.39	87.98	9.25	59.26	99.87	68.66
Room	bL6	2.41	53.20	4.31	86.37	10.96	70.21	101.60	69.85
Raw	oR8	4.97		4.97		16.37		153.21	
Refrig.	cR7	2.00	40.24	4.85	97.59	9.93	60.66	102.80	67.10
Refrig.	cR9	2.03	40.85	5.15	103.62	10.23	62.49	110.10	71.86
Raw	oL8	5.00		5.22		17.23		154.64	
Water	dL7	1.94	38.80	3.77	72.22	7.62	44.23	103.10	66.67
Room	bL9	2.27	45.40	4.22	80.84	9.89	57.40	92.46	59.79
F Values for Differences Between Thawing Methods		1.76		2.50		12.42*		2.03	

* Significant.

TABLE 4

Vitamin Content of Meat and Drippings

Thiamine µg/g				Riboflavin µg/g				Pantothenic Acid µg/g				Nicotinic Acid µg/g			
73° C.	Room	Refrig.	Water	73° C.	Room	Refrig.	Water	73° C.	Room	Refrig.	Water	73° C.	Room	Refrig.	Water
4.81	4.01	4.07	3.60	5.94	5.99	6.89	6.94	10.56	13.74	13.88	11.23	122.84	176.58	128.19	102.55
4.30	4.94	4.86	3.71	6.87	5.95	7.06	6.93	12.68	13.36	14.66	12.66	140.51	171.75	107.91	115.34
4.84	4.99	6.61	3.28	7.64	5.82	7.30	6.47	12.03	14.26	12.95	11.74	127.60	154.00	183.82	124.23
4.58	5.08	5.08	5.49	7.07	7.44	6.48	7.26	13.11	15.49	16.28	11.35	155.25	144.20	197.97	172.90
4.37	4.72	4.63	3.43	6.67	7.22	6.97	6.94	12.18	13.17	17.79	11.15	166.02	175.84	189.86	146.02
4.15	4.79	3.98	4.65	6.72	6.93	6.11	6.95	11.64	15.34	12.66	12.19	151.02	129.90	168.81	179.62
3.77	4.43	3.46	3.88	5.33	5.65	6.46	5.07	12.15	18.25	13.21	10.63	155.42	157.98	151.11	122.46
3.73	3.52	3.21	3.07	5.22	5.13	5.84	4.80	15.33	16.09	12.62	12.10	164.77	130.11	148.87	141.85
4.36	3.00	3.06	2.99	4.90	5.26	6.00	4.60	14.77	13.35	12.50	12.06	151.22	111.16	147.47	132.79
F Values for Differences Between Thawing Methods															
3.89*				1.31				7.48**				2.11			

* Significant. ** Highly significant.

percentage retention of the vitamins than those thawed at room or refrigerator temperature. However, none of the thawing methods resulted in large differences in vitamin retention. The percentage of riboflavin retained in the cooked steaks appears to be abnormally high. It may be that the cooking process aids in the formation of riboflavin from its precursors or liberates it from tightly bound molecules, as Hinman, Tucker, Jans and Halliday *(4)* have reported excessively high riboflavin retention in braised beef. It is difficult to explain the higher retention of riboflavin in the steaks from animal 1, since all the samples were treated in the same manner.

In an attempt to determine whether the vitamins were actually lost or destroyed in the cooking process or were extracted from the meat during braising, the drippings from the meat were analyzed. Since both the meat and the drippings are usually consumed, it seemed logical to consider the total content of the vitamins from both of these sources. The results are shown in Table 4. The statistical analysis showed no significant differences in the content of riboflavin and nicotinic acid between the different methods of thawing. There were significant differences in the thiamine content between the different methods of thawing. The analysis of variance showed that the samples thawed at 73° C., at room temperature and in the refrigerator retained a significantly greater amount of thiamine in the meat and drippings ($P<.05$) than those thawed in running water.

Highly significant differences in pantothenic acid content were found between the different thawing methods. The analysis of variance showed that thawing in the refrigerator and at room temperature produced no significant differences. Thawing at room temperature gave significantly higher values than thawing either at 73° C. ($P=.05$) or in running water ($P<.01$). Thawing in the refrigerator also produced a significantly higher value than thawing in water ($P<.01$). When the pantothenic acid content of both the meat and drippings was considered, thawing at room temperature or in the refrigerator allowed better retention than thawing at 73° C. or in running water. Thawing in running water produced the greatest losses of this vitamin.

When the total retention of the vitamins in the meat and drippings was considered, the steaks thawed in water showed the greatest loss. This undoubtedly was due to a leaching out of the vitamins during the thawing process. The leaching out of other substances as well as the vitamins may have had an effect upon the palatability since the desirability of the flavor was also decreased in these steaks.

Summary

The palatability and vitamin content of the frozen round steaks from 3 beef animals, thawed by 4 different methods, were determined. The steaks were thawed in the refrigerator, at 73° C., at room temperature and in running tap water. After thawing, the steaks were cooked by braising and each compared in vitamin content with the adjacent steak which had been analyzed in the fresh state.

Thawing the steaks in water decreased the desirability and intensity of the flavor of the fat, and the quality and quantity of the juiciness of the meat.

Thiamine, riboflavin, pantothenic acid and nicotinic acid values were determined on the steaks and drippings. Statistical analyses of the data showed no significant differences between the methods of thawing for the thiamine, riboflavin and nicotinic acid content, but there was a significant difference in the pantothenic acid values between the steaks thawed in the refrigerator and at room temperature and those thawed in water. The retention was greatest in those thawed in the refrigerator.

When the total retention of the vitamins in both the meat and the drippings was considered, the losses were greater when the steaks were thawed in water than by the other methods. Thawing at room and refrigerator temperatures gave slightly better retention than thawing at 73° C.

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Effect of 0° F. and 15° F. Storage on the Quality of Frozen Cooked Foods[a]

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Three frozen precooked meals were stored at 0° and 15°F. for three months and sampled at bi-weekly intervals. In a subsequent experiment, improved meals of the same kind were stored six months and judged each month. Factors tested were palatability, peroxide number of gravy, ascorbic acid of broccoli, bacterial content. Foods stored at 15°F. lost palatability sooner than those stored at 0°, with certain exceptions. General suggestions for the preparation of successful precooked meals are included.

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The meals studied were:

1. Chicken paprika and gravy
   Mashed, orange-flavored sweet potatoes
   Broccoli
2. Beef roulades and gravy
   Cottage fried potatoes
   Mashed rutabagas
   Spiced cherries
3. Beef pot roast and gravy
   Baked stuffed Idaho potatoes
   Buttered snap beans
   Cinnamon pears

Procedure

Food preparation. The suggestions and recipes given in the bulletin "Foods from the Freezer: Precooked or Prepared," by Fenton and Darfler *(3)*, were used as a basis for the procedures employed in the preparation of the meals.

Sufficient foods were prepared in one batch to provide portions for judging throughout the storage period. On the basis of preliminary work, the size, shape, and degree of doneness for each serving of food was controlled so that after a 15-minute reheating period it was at serving temperature and yet not overcooked.

Since traces of iron and copper tend to hasten rancidity development as well as catalyze the oxidation of ascorbic acid, no copper or iron utensils were used with the exception of the Dutch ovens used in cooking the beef roulades and the pot roast.

Space will not permit giving details of the preparation of the foods. However, variations from standard procedures are included in this report since many of them were factors in the storage life of the foods.

Chicken paprika was prepared essentially as outlined for "smothered chicken" by Rombauer *(7)*.

Gravy. Supplementary studies showed that: (1) an excess of fat tended to cause separation of the gravy, (2) blending the gravy in a Waring Blendor for 2 minutes helped prevent separation, but overblending resulted in a thinned, bubbly gravy, and (3) that the addition of a small amount of raw flour waterpaste to gravy that had been completely cooked and then cooled helped to prevent separation during reheating and no undesirable raw starch flavor resulted and also (4) that cooling the cooked gravy could be hastened by adding frozen stock to it and by placing the container in ice water.

The gravy used in the second storage study, therefore, as well as that for the beef roulades and the pot roast was prepared as follows: The fat was removed from the pan gravy. For each cup of liquid (stock and fat-free pan drippings), one tablespoon of fat and two tablespoons of flour were used. The fat and three-fourths of the flour were blended and cooked, then two-thirds of the liquid was added and the gravy was completely cooked. It was chilled by adding the remaining stock that had been frozen, and then this mixture was blended with the rest of the flour in a Waring Blendor.

Broccoli of the Calabrese variety was harvested in the evening and stored overnight in the hydrator of an electric refrigerator. That for both storage lots was scalded for 3 minutes and chilled in ice water as is commonly done prior to freezing. In preliminary studies,

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broccoli that had been cooked beyond the scalding stage was overcooked after the 15-minute reheating of the meal. Broccoli for the first storage lot was cut into 3½-inch lengths. Stalks having a diameter of more than 1 inch were split. In order to obtain a closer pack broccoli for the second storage study was cut into 3 by ½-inch pieces. Butter and salt added in the first study developed an off-flavor; therefore, in the second study they were not added until just before the frozen broccoli was heated.

Both lots of *sweet potatoes* were made with fresh orange juice because it resulted in better retention of flavor and color during storage than did milk, Fenton and Darfler *(3)*. Care was taken not to incorporate air during puréeing.

Beef roulades were made by rolling strips of smoked bacon inside thin slices of round of beef and braising them.

The *cottage fried potatoes* were New York State Irish Cobbler potatoes that had been sliced 1/8 to 1/16 inch thick, immersed in water, drained and sautéed in hydrogenated vegetable shortening.

Spiced cherries were made from canned, sweetened, sour pitted cherries. The cinnamon pears were also made from canned fruit.

The *stuffed baked potatoes* were prepared by the standard method using Idaho potatoes. The skins were first lightly greased to avoid having a hard dry skin. Grated process cheese and pakrika were sprinkled over the stuffed potatoes just before they were lightly broiled.

The *green beans* (commercially frozen) were partially cooked and then seasoned with butter, salt, and pepper.

Packaging. All the foods were packaged in individual portions. Those for the first storage study of Meal 1 were placed in aluminum foil molds, frozen, then sealed in moisture-vapor-resistant cellophane. Molds, 5½ by 3½ inches, were each filled with 1 piece of light and 1 piece of dark chicken meat and ¼ cup of gravy. Molds, 2½ by 3½ inches, were filled to a depth of ½ inch with either broccoli or mashed sweet potatoes. In the second storage study of Meal 1, the gravy was increased to ½ cup to prevent drying of the chicken. Each serving of chicken weighed about 7½ ounces. The broccoli, about 2¾ ounces, and mashed sweet potatoes, 4½ ounces, were packaged in aluminum foil molds. The other foods were frozen in large, shallow, aluminum pans, cut into servings, and sealed in cellophane. For convenience, each complete dinner was packed in a plastic-lined paper bag.

Freezing. The foods were frozen in the freezing compartment of a home freezing cabinet that registered —20° F. (—29° C.) near the bottom of the compartment. A fan was used to circulate the air.

Storage. Meal 1. In the first storage study of this meal, half of the foods were stored at —6° to —2° F. (—21° to —19° C.). The other half was stored at —5° to 8° F. (—20.5° to —13.5° C.) over a 1¼ hour cycle, for the first month because of difficulty in regulating the temperature; and at —3° to 18° F. (—19.5° to —8° C.), over a 2½-hour cycle, for the remaining two months. Portions of these foods were removed from the two storage boxes for palatability tests at the end of 20, 55, 79, and 93 days.

In the second storage study of Meal 1 half of the foods was stored at —6° to —2° F. (—21° to —19° C.), and half at 13° to 15° F. (—10.5° to —9.5° C.) Foods from each storage temperature were judged at monthly intervals.

Meals 2 and 3. Half of the foods were stored at —6° to —2° F. (—21° to —19° C.), and half at 13° to 15° F. (—10.5° to —9.5° C.). Portions of Meal 2 were removed for palatability tests after storage at the lower temperature for four days; and from both storage temperatures after storage for 21, 35, 49, 63, 77 and 86 days. Portions of Meal 3 were judged for palatability after storage for 7 days at the lower storage temperature; and from both storage temperatures after storage for 25, 29, 53, 67, 81, and 87 days.

For clarity, the lower storage temperature will be referred to as 0° F., the higher temperature as 15° F., throughout the paper.

Reheating. The foods were unwrapped and placed on glass pie plates that were divided into sections with strips of aluminum foil to keep the foods from running together. All of the plates were covered partially with aluminum foil; air spaces of ¼-inch were left at two sides of the foil to allow the hot air to circulate easily over the food except in the case of the cottage fried potatoes which were left half covered. If no foil was used the potatoes were hard and dry; if completely covered they were soggy.

The solidly frozen foods from the first storage lot of Meal 1 were reheated for 25 minutes in a Despatch oven (375° F.). The foods from the other meals were placed in a preheated Maxson Whirlwind oven (300° F.) and heated for 15 minutes.

The cinnamon pear wedges were thawed at room temperature while the meal was being heated.

Palatability. The foods were scored for palatability by a panel of trained judges chosen chiefly on the basis of their interest in the problem and on their ability to concentrate. The panel varied from 8 to 12 people and consisted of half men and half women.

When a new menu was tried, it was judged as a whole for combinations of colors, shapes, textures, and flavors. Each of the four factors was scored on the basis of 100 for a perfect combination.

In addition, at each storage period, each food was judged separately for appearance; flavor, including taste and aroma; texture; and moisture. Two dinners, the same meal from each of the two storage temperatures, were judged together. In this way the judges scored four or five paired foods at one time. Each food was coded and the judges were not told how it had been treated.

The score cards were made as simple as was consistent with obtaining the desired information. Each of the four factors of palatability (appearance, flavor, texture, and moisture) was scored as perfect 100, excellent 80, good or average 60, fair 40, poor 20 or unappetizing 0. The judges were directed to place a score in between any two of the above six categories, when they thought it suitable. If the food was scored low,

TABLE 1

The Effect of Storage of 0° F. and at 15° F. on the Total Palatability Scores of Three Frozen Cooked Meals

Meal No.	Total Palatability Scores [1]							
	Before Storage	Stored 3 Months				Stored 6 Months			
		0° F.		15° F.		0° F.		15° F.	
		Score	Change	Score	Change	Score	Change	Score	Change
1. First lot	70	62	— 8	58	—12	....		....	
Second lot	82	77	— 5	79	— 3	76	—6	67	—12
2.	67	64	— 3	55	—12	....		....	
3.	71	59	—12	60	—11	....		....	

[1] Average of scores for appearance, flavor, texture, and moisture of all the foods comprising the meal. A perfect score was 100; excellent, 80; good-average, 60; fair, 40; poor, 20; and unappetizing, 0.

the judge was requested to give the reason. The total palatability score for a food was the average of the scores for the four factors. The total palatability score for a meal was calculated by averaging the total scores of each of the foods comprising the meal.

All of the foods were judged at noon just before lunch. The judges first noted the over-all appearance of the foods, and then they took portions of each food to score. The judges were widely separated and no conversation occurred during the judging.

Chemical analyses. Determinations of the peroxide numbers of the fat in the chicken gravies were made by the method outlined by Volz and Gortner *(8)*. Ascorbic acid determinations were made by the indophenol-xylene extraction method of Nelson and Somers *(5)*.

Bacterial analyses. The bacteria found in foods from Meal 2 and 3 were classified according to the *Manual of Determinative Bacteriology (1)*. The plate counts of the foods at the start and at the end of three months of storage at the two temperatures were made by a standard method *(2)*.

Results

The total palatability scores of the three frozen meals at the start and at the end of storage at the two temperatures are given in Table 1. Keeping in mind that 60 represented average good quality one can see that Meals (first lot) 2 and 3 scored good plus before storage. The second lot of Meal 1 scored higher chiefly because of the gravy which rated 30 points higher than that in the first lot. It was one-third higher in both appearance and consistency.

At the start of the storage studies the frozen foods scoring excellent or better were chicken paprika (second lot), mashed sweet potatoes (both lots), broccoli (second lot), cinnamon pears, and baked stuffed potatoes. Foods scoring good plus were chicken paprika (first lot), broccoli (first lot), chicken gravy (second lot), mashed rutabagas, spiced cherries and pot roast gravy. Foods scoring good were beef roulades and gravy, pot roast, fried potatoes, and green beans. The first lot of chicken paprika gravy scored fair plus.

The effect of storage at the two temperatures on the individual quality factors will be discussed under each meal. Only a few typical changes will be presented in the accompanying Figures because of lack of space. These are of foods in Meal 1, Lot 2.

Meal 1. First lot. After storage for three months, the foods stored at the higher temperature had lost palatability slightly more than those stored at the lower temperature. Those stored at 0° F. rated average good and those stored at 15° F. rated slightly lower (Table 1). The appearance and the flavor of the chicken were the only factors which had not decreased. These remained about the same during storage at both temperatures. The most marked losses occurred in the (a) texture of the chicken stored at 15° F. (the skin had become tough); (b) flavor of the gravy stored at 15° F. (described as "stale"); (c) moisture of the sweet potatoes stored at 15° F. (slightly dry); (d) flavor of the broccoli stored at both temperatures (probably due to an off-flavor of the butter). At the end of 2½ months of storage, however, the broccoli stored at 0° F. was still maintaining a "good flavor."

Meal 1. Second lot. After 3 months' storage a slight drop in palatability was noted at both temperatures (Table 1).

At the end of 6 months' storage, the score for the meal stored at 0° F. remained about the same as at the end of 3 months of storage; the score for the meals stored at 15° F., however, decreased.

Trends in losses of specific palatability factors at the two storage temperatures are given in Figure 1. The chicken stored at 0° F. lost flavor and moisture. The chicken stored at 15° F. lost these two factors slightly more and also texture. That the loss in texture for this second lot of chicken was not so great as for the first lot was probably because of its better covering of gravy.

The gravy gradually scored lower in flavor, texture, and moisture, but the appearance remained about the same. The losses were slightly greater and occurred sooner in the gravy stored at 15° F. than in that stored at 0° F.

No peroxide development was noted in the chicken gravy after storage for 5 months at either temperature. Gravy from the first storage lot likewise showed no peroxide development after storage for one year at 0° F. Failure to develop rancidity may be partially due to a low fat content. Furthermore, care had been taken not to overheat the fat during preparation and to avoid any contact with iron or copper. It is possible, too, that the paprika may have decreased any tendency toward rancidity.

Of all the foods in the meal, the sweet potatoes retained their palatability best. Little if any loss of quality occurred during storage at 0° F. This may be attributed at least partially to the use of orange juice and also to the solid pack that was possible with the puréed vege-

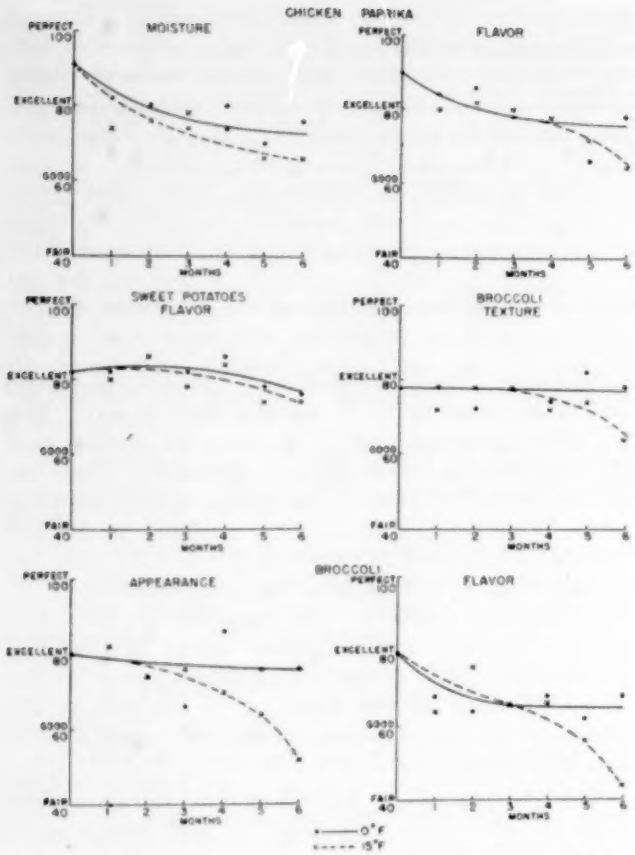

Fig. 1. EFFECT OF STORAGE ON SOME FACTORS IN THE PALATABILITY OF MEAL 1, LOT 2.

table. These potatoes retained their palatability to a slightly greater extent than did those from the first storage lot, probably because they were packaged better. Changes in flavor scores are shown in Figure 1.

During storage of the second lot of broccoli for 6 months at 0° F. palatability losses were not severe. Nor were they great from the broccoli stored at 15° F. after 3 months but at the end of 6 months the broccoli was markedly lower in appearance and flavor and slightly lower in texture and moisture. The slight loss of palatability in the broccoli stored at 0° F. for 6 months was due mainly to a drop in the score for flavor (Figure 1). This lot of broccoli held up better than did the first lot. Tighter packing, better packaging, and the absence of butter and salt probably all contributed to the longer storage life of the second lot.

After 5 months of storage at 0° F. the broccoli (second lot) contained 50 mg. of ascorbic acid per 100 grams and the broccoli stored at 15° F. contained 20 mg. of ascorbic acid per 100 grams, or only two-fifths as much. During heating of broccoli from both storage temperatures in the Maxson oven, similar amounts of ascorbic acid were retained (83 and 77 percent). During storage for 3 months at 0° F. little, if any, drop in the total palatability scores of Meal 2 occurred (Table 1). Even after storage for 7 months at 0° F. no loss of palatability was observed in any of the foods. The use of smoked bacon in the roulades, the fat sealed and slightly dry surface of the potatoes, and the solid pack that was possible for the mashed rutabagas probably contributed toward retention of palatability.

At the end of 3 months, all of the foods stored at 15° F. scored appreciably lower than those stored at 0° F. During the 15° F. storage, the roulades and the gravy gradually lost flavor, texture, and moisture. The rutabagas held up well until the last 2 weeks of storage when the yellow color became slightly dull and the flavor stale.

The cherries stored at 15° F. scored lower in all four factors than did those stored at 0° F., which were very acceptable even after storage for 7 months. The cherries stored at the higher temperature were shriveled and the skins slightly tough.

Meal 3. The total palatability score for Meal 3 after storage for 3 months dropped to the same extent at both temperatures: from good plus to an average of good (Table 1). Losses occurred in appearance, flavor, texture, and moisture.

During storage at both temperatures, the pot roast gradually lost appearance and moisture.

A marked drop occurred in the score for the gravy during the first 3 weeks of storage. Although all four factors scored lower, the most severe losses occurred in appearance and moisture.

The baked stuffed Idaho potatoes decreased during storage from almost excellent to good. Although a loss was noted in each factor, the most severe loss occurred in texture. A cottony texture was noted after two months of storage at 15° F. and after about 11 weeks of storage at 0° F.

The green beans scored almost good at the start of the study and about fair after 3 months of storage. Their color was poor and they were dry after reheating. The length of storage prior to their arrival in the laboratory was not known.

The cinnamon pear wedges scored excellent. They retained their palatability remarkably well even at the higher storage temperature. This was probably because they were canned and therefore all enzymes had been inactivated and they were practically sterile. Their bright color, refreshing flavor, and cold temperature made them a very appetizing addition to the reheated meal.

Bacterial analyses of the foods in Meal 2 and 3 showed the presence of bacilli, staphylococci, and some achromobacter (Table 2). Ten days after preparation and freezing, the foods contained well under the 10,000 to 100,000 per gram allowance (or higher depending upon the food) that has been suggested as permissible by Fitzgerald *(4)* for frozen cooked foods. During storage for 3 months at both temperatures the number of bacteria decreased. Likewise, Proctor and Phillips *(6)* found a decrease in the plate count of microorganisms in frozen precooked foods during storage at —5° F. (—20.5° C.). The number of bacteria present was about the same in the foods stored at each of the two temperatures. This is confirmation of the well-accepted finding that bacteria do not grow at 15° F. or below. Neither the spiced cherries nor the cinnamon pears contained any bacteria. This may be partially due to their relatively high sugar content and to the fact that they had been canned.

TABLE 2

Analyses and Bacterial Contents of Two Precooked Frozen Meals

Food	Stored 10 days at 0° F.		Stored 2 months at 0° F.	Stored 3 months at 15° F.
	Types	Plate Count per gram	Plate Count per gram	Plate Count per gram
Meal 2				
Beef roulades and gravy	mostly bacilli; some staphylococci	6200	2500	2300
Cottage fried potatoes	mostly bacilli; some staphylococci	1140	150	320
Mashed rutabagas	all bacilli	6100	3600	3600
Spiced cherries	.....	0	0	0
Meal 3				
Pot roast and gravy	mostly bacilli; some staphylococci	6800	2500	3000
Baked stuffed potatoes	mostly bacilli; some achromobacter; some staphylococci	1700	2700	2200
Green beans	all staphylococci	570	610	.....
Cinnamon pears	.....	0	0	0

General Suggestions

The following suggestions are made for the preparation of successful frozen precooked meals:

1. Choose an outstanding menu from the viewpoint of combination of colors, shapes, textures, and flavors. Make sure that each food item is of excellent quality.

2. Control the degree of cooking so that all the foods to be heated together will be cooked to optimum doneness and heated to serving temperature at the same time.

3. Control the size and shape of the servings of each food so that it will neither dry out during reheating nor remain frozen in the center. If compartmented plates are to be used for reheating, freeze the foods in containers of corresponding dimensions and shapes.

4. Pack foods solidly as possible to avoid air spaces. This will help to prevent the formation of cavity ice, drying of the foods, and oxidative changes. In order to pack some pieces of meats and some vegetables solidly, sauce may be advisable to fill the air spaces. Puréed vegetables can be packed solidly without sauce. The relatively longer storage life of the second lot of chicken paprika, which was amply covered with gravy, of the second lot of broccoli, and of the mashed sweet potatoes and rutabagas may be partially attributed to solid packing.

5. Reheat the portions of foods in containers of a size and shape similar to the foods themselves. This will help to (1) prevent different foods and sauces from mingling, (2) keep the edges of a sauce or gravy from spreading as it thaws and thus being overheated and (3) prevent the food itself from becoming dry.

6. Handle all foods in a sanitary manner. Chill them quickly, package them carefully, avoid unnecessary transfer during preparation, freeze them promptly, and reheat them quickly. The addition of frozen stock or water to a thick gravy helps to cool it rapidly.

7. Do not expect frozen cooked foods to retain their initial high quality during long storage. Store at least as low as 0° F. if for more than one month.

8. All left-overs are not adaptable to freezing. Some will be overcooked when they are reheated. Unless the foods are chilled and frozen rapidly, the bacterial content is likely to be high. The freezing of cooked foods should be planned.

9. If several foods, put into the freezer at the same time are to be used in one meal, they should have approximately the same storage life; or the storage period of the meal should be limited by the food having the shortest storage life.

10. To help prevent sauces from breaking during reheating, reserve from ¼ to ⅛ of the starch and add it to the chilled gravy before freezing. Blending the gravy in a Waring Blendor for not over 2 minutes helps to make it smooth. Overblending results in the incorporation of too much air, a foamy sauce, and often a thinned sauce.

11. Do not use an excess of fat as it tends to separate. An easy way to separate fat from hot gravy is to use a large separatory funnel with a large drainage spout.

12. Cover foods with aluminum foil during heating to the extent necessary to avoid overdrying. Some foods such as fried potatoes and pastry turnovers may become soggy, however, if completely covered.

Summary

Three meals were prepared in quantity, frozen at —20° F. (—29° C.), packaged in single servings, and stored at two temperatures. In all of the storage studies, the lower temperature ranged from —6° to —2° F. (—21° to —19° C.); in the first storage study, the higher temperature ranged from —3° to 18° F. (—19.5° to —8° C.); and in the other three storage studies, from 13° to 15° F. (—10.5° to —9.5° C.). At approximately bi-weekly intervals over a period of 3 months, the foods from Meal 1, 2, and 3 were judged by a panel of from 7 to 12 judges. The following year a second lot of Meal 1 was judged at monthly intervals over a period of 6 months.

Supplementary studies were made of methods of preparation of some of the foods used in the above meals.

Analyses for peroxide number of chicken gravy and for ascorbic acid content of broccoli were made.

Analyses were made of the types of bacteria in two of the meals and plate counts of these same frozen meals were made before and after storage for three months at each of the two temperatures.

The foods that retained their original palatability best during storage were the sweet potatoes and the cinnamon pears stored at both temperatures; and the ruta-

bagas, the second lot of broccoli, and the beef roulades, stored at 0° F. (—18° C.). The foods that retained their palatability fairly well were the chicken paprika (first lot), chicken gravy, beef roulades gravy, fried potatoes, and the pot roast. The foods that lost palatability to the greatest extent were the broccoli (first lot), the spiced cherries, the pot roast gravy, the stuffed potatoes, and the green beans. Although the second lot of chicken paprika dropped in palatability, the original score, above excellent, was so high that the final score even after storage for 6 months at 15° F. (—9.5° C.) was half way between good and excellent.

The deleterious effect of the higher storage was very marked after three months storage of most of the foods.

Along with better retention of palatability, broccoli stored at 0° F. (—18° C.) contained about two and one-half times the ascorbic acid as did that stored at 15° F. (—9.5° C.). The bacterial contents of foods stored at both temperatures were low, primarily because initial contents were low.

Acknowledgment

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Electrochemical Evaluation of Organic Coatings for Food-Container Closures

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A technique for accelerated testing of coated metal closures on glass food containers is described. Failure of the closures by pitting and perforation, which occurs during protracted storage under normal conditions, can be duplicated in a few hours. On the basis of the results obtained in the use of this test, it is suggested that corrosion of the metal may be due mainly to local concentration cell effects, with the food product acting as the electrolyte and with the metal exposed through pores in the organic coating acting as anodes and cathodes. The anodic areas tend to produce perforation of the metal. The possible mechanics of the formation of these concentration cells and the factors limiting their continued functioning toward perforation of the metal are discussed.

Introduction

Coated metal closures on glass containers packed with certain processed foods have been known to perforate during storage in warehouses or on the shelves in retail stores. In the organic coating on the interior surfaces of the perforated closures, there were circular blue spots approximately ⅛-inch in diameter. Removal of the coating showed that the metal had been detinned under the blue spot and, in the center of the detinned area, there was usually a pit in the metal. Some of the pits were sufficiently deep to be perforations.

By the technique developed for accelerated testing, this type of failure can be reproduced in the laboratory in a few hours. This technique is described in this paper; and, on the basis of results obtained, the cause and cure of perforation is suggested.

Test Procedure

Metal closures are usually fabricated from flat sheets of tin-plate roll-coated on one side with a sanitary lining [b] and on the other side with a lithographed, decorative and protective coating. Test panels (approximately 3″ x 6″) are cut from these sheets; and each test panel is cut in half (3″ x 3″). One end of a glass cylinder (3 inches long, 2 inches outside diameter, 1/16-inch wall thickness, and open at both ends) is then sealed with wax to the coated face of one half of the test panel. The resulting cup is filled to within ¼-inch of the top

[a] Contribution from the Stoner-Mudge, Inc. Multiple Industrial Fellowship on Protective Coatings.

[b] The term "sanitary lining," as used here, is intended to denote any organic coating which is free of all toxic ingredients and which imparts no taste or odor to the packaged food. The color of the coating is not restricted.

with a suitable electrolyte, and the coated side of the other half of the same test panel is sealed with wax to the remaining open end of the glass cylinder. (The electrolyte selected was an aqueous solution of 15% sodium chloride and 1% malic acid.) The assembly is then laid on its side so that both halves of the panel are vertical and are in contact with the electrolyte. This cell is called the "perforation-test cell."

One cell (approximately 2 volts) of a lead storage battery is connected across the two plates of the perforation-test cell. One plate is thus made anodic, and the other plate cathodic, with respect to each other. After approximately 16 hours, the perforation-test cell is taken apart and the two panels are rinsed in tap water and dried with a towel.

On the panel that was anodic in the perforation-test cell there is a number of blue spots in the coating that was exposed to the electrolyte; and at the center of each blue spot there is a pit in the metal. Some of these pits may be sufficiently deep to perforate the metal. This condition is similar to that observed on metal closures on food jars after a storage period. The panel that was cathodic will not have any blue spots or pits, but it will have areas on which the coating is adhering loosely to the metal surface. The loosened coating is removed by pressing Scotch Cellulose Tape on the coating surface and then pulling the tape off abruptly. Under this loosened coating, the surface of the metal is bright and uncorroded. This condition usually can be found on perforated closures on food jars.

The amount of coating which remains on the panel is estimated and rated by the following scale:

10 = coating intact (100% remaining on panel),
9 = slight evidence of removal of coating,
8 = definite evidence of removal of coating,
7-1 = increasing amounts of coating removed,
0 = coating completely removed.

Fig. 1 is a photographic reference standard for evaluating the amount of coating which remains on the panel. The approximate relationship between the rating scale and the actual percentage of coating which remains on the panel is shown in Fig. 2. The rating scale is used because actual measurement of the area of coating remaining is difficult and time-consuming.

The number of blue spots on the panel which was anodic in the test cell and also the number of perforations are counted and recorded.

Experimental

A large number of test panels coated with two different coating systems were evaluated by this test method. The results, indicated schematically in Fig. 3, show that there are definite relationships between the amount of coating which remains on the cathode panel, the number of blue spots on the anode panel, and the number of perforations of the anode panel.

Another group of experiments was made with a duplicate set of panels, to study the effect of concentration of sodium chloride and malic acid in the electrolyte. These results are recorded schematically, in Fig. 4. It will be noted, in Fig. 4, that the amount of coating

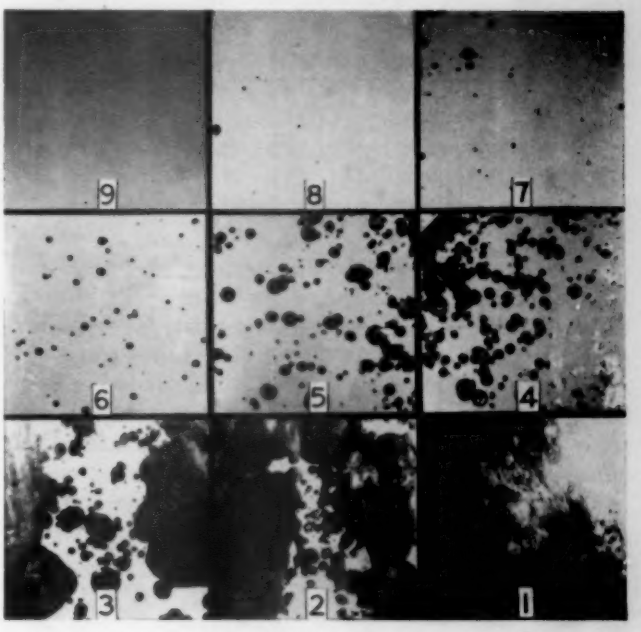

Fig. 1. Rating scale for estimating amount of coating remaining on cathode.
10 = All remaining
0 = None remaining

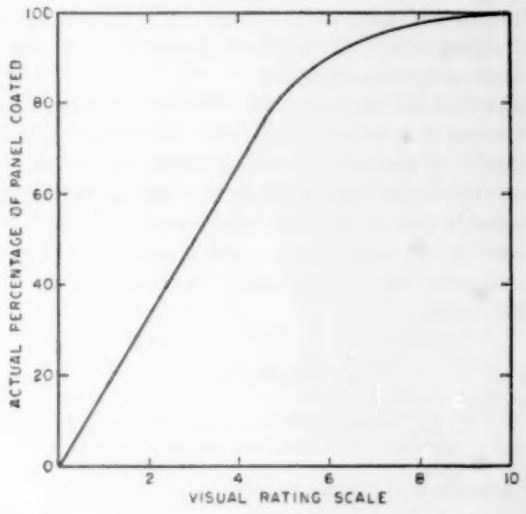

Fig. 2. Relationship between rating scale and actual percentage of panel coated.

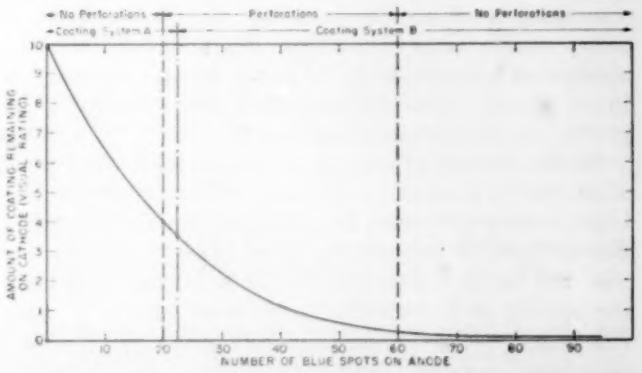

Fig. 3. Relationship between perforations, amount of coating remaining on cathode and number of blue spots on anode.

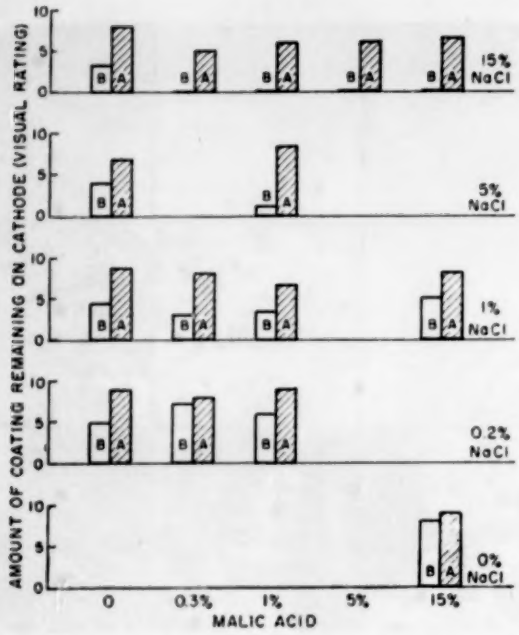

FIG. 4. Effect of concentration of electrolyte upon amount of coating remaining on cathode. (Comparing coating systems A and B).

which remains on the cathode panels coated with system "A" is, in all cases, greater than that remaining on the panels coated with system "B." The blank spaces not designated by "A" or "B" indicate that no tests were run at these concentrations.

The effect of the length of time of electrolysis and of the amount of applied voltage were investigated also, but the results of that study are not reported in detail here. It was found that the amount of coating remaining on the cathode panels was inversely proportional, and the number of perforations of the anode panels directly proportional, both to the time of electrolysis and to the applied voltage.

Discussion

The following mechanism is proposed in explanation of the perforation of coated tin-plate closures:

1) *Formation of "Blue Spots"*

The electrolyte—i.e., the food product—attacks the metal at a discontinuity in the organic coating. If the electrolyte is such that the tin is cathodic to the iron, the tin will be protected and the iron will corrode at a somewhat accelerated rate (Fig. 5a). This type of attack usually takes place in the presence of air or in the absence of organic acids. The tin layer frequently is intact except for a small original discontinuity. The cavity, or pit, is beneath the tin layer.

In the absence of air and in the presence of organic acids, the tin is anodic to the iron. When the electrolyte is in contact with both the iron and the tin through a discontinuity in the coating, under these conditions, the iron will be protected and the tin will corrode beneath the coating at a somewhat accelerated rate. The area that is detinned will appear as a blue spot under a white-pigmented organic coating (Fig. 5b). As detinning proceeds, the space between the coating and the iron surface is filled with corrosion products and the tin is no longer capable of protecting the iron at the center of the discontinuity in the coating. Corrosion of the iron then takes place. If the iron and tin salts formed are insoluble, the corrosion reaction will be retarded and eventually may stop entirely. However, in the presence of many organic acids, such as malic acid, these salts are soluble and corrosion can continue. A pit will then form at the approximate center of the blue spot (Fig. 5c). If the layer of tin were not present initially, the iron would be attacked immediately at the discontinuity in the organic coatting and a pit would be formed. Corrosion would occur at a discontinuity, regardless of the type of organic coating involved; but, if no other corrosion mechanism were operating, it would require a very long time to cause perforation of the metal.

2) *Development of Perforations*

Unpublished experiments have shown that metal coated with an organic coating will corrode at gross discontinuities in the coating at a greater rate than uncoated metal. In other words, metal exposed at discontinuities in organic coatings (all of which are imperfect barriers to moisture or to ions) is anodic to the coated metal. This is probably due to a "concentration-cell effect," i.e., difference in concentration between the electrolyte in contact with the metal through a discontinuity in the coating and the electrolyte in contact with the metal through the permeable organic coating. The same experiments, run on glass coatings which can be perfect barriers to moisture or to ions, did not show this effect.

Fig. 5d illustrates a section of coated tin-plate on which there are two discontinuities, one smaller than the other. Because of the difference in size of the two,

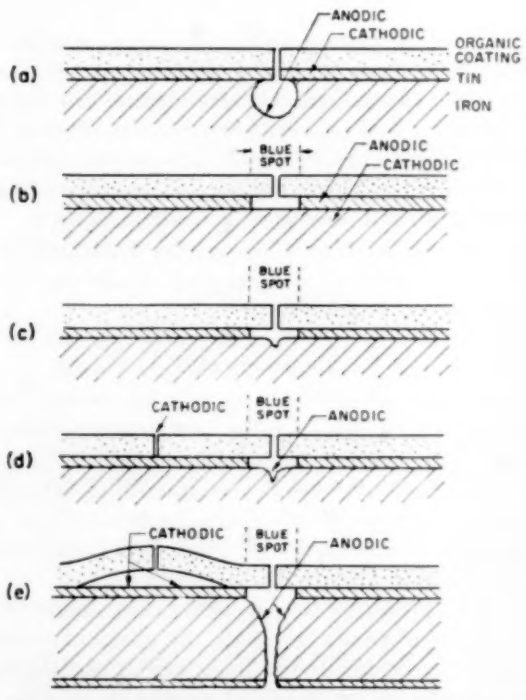

FIG. 5. Schematic sketch of perforation mechanism.

the concentration of the electrolyte in contact with the metal surface through these two discontinuities will differ if one or both of them restrict the free flow of electrolyte. The larger of the two will permit a higher concentration of electrolyte to contact the metal surface and thus, by a concentration-cell effect, make that area anodic and the area of the smaller discontinuity cathodic *(3)*. An electrolytic cell is formed; corrosion occurs at the anode area; at the cathode, alkali is formed as a result of electrolysis of sodium chloride in the solution. If both the anode and the cathode areas are held constant and if the reaction products are not removed as fast as they are formed, the rate of corrosion will decrease. This reaction will thereby cause some additional pitting of the metal.

Because most corrosion reactions of this type are under cathodic control, the rate of corrosion can be increased by increasing the cathode area. If the bond between the organic coating and the metal at the cathode area is resistant to deterioration by the alkali formed at the cathode, no further increase in cathode area occurs. However, if the bond is degraded by alkali formed at the cathode, so that the coating is loosened, the cathode area is enlarged and the rate of corrosion at the anode increases until the metal is perforated (Fig. 5e).

The perforation-test cell makes it possible to investigate and measure the extent to which the bond of an organic-coating system to a metal surface is resistant to degradation by the alkali formed at a cathode during the corrosion reaction. It has been found that the bond is dependent upon the type of organic coating, the method of application, the cleanliness of the metal surface coated, the baking schedule, and other factors.

Modification of the Perforation-Test Cell

A further modification of the perforation-test cell has been made so that it can be used as a quality-control test for commercially coated sheets of tin-plate. Instead of using glass cells as test cups, which limited the area of coating investigated, a procedure was devised for testing panels of any size up to a full sheet of tin-plate, as follows:

A margin ½-inch wide along each edge of both sides of a test panel is coated with melted wax; and the unwaxed portion of the panel is covered with a waxed-edge blotter which is saturated with an excess of electrolyte (15% sodium chloride plus 1% malic acid). A duplicate test panel cut to the same size as the first panel and similarly waxed is laid with its coated face against the blotter. This assembly, or test cell, then consists of two duplicate coated panels separated by a blotter saturated with electrolyte. Several such cells may be piled, one on top of the other. A flat weight is then placed on top of the cells. Each pair of coated panels separated by the electrolyte-saturated blotter—i.e., each cell—is connected in parallel to one cell of a lead storage battery (2 volts), and the current flow between each pair of panels is tested to make sure of electrical contacts and to find and eliminate any accidental short circuits. The specific gravity of the battery electrolyte also is measured, before and after the test, to detect any short circuits or excessive current drain during the test. The electrolysis is run for 16 hours; and at the end of this time the current flow is checked again. The test cells are then taken apart, and each panel is rinsed with running tap water and dried with towels. The coating loosened by the test is removed immediately with cellulose adhesive tape.

The panels are rated for the amount of coating which remains on the cathode, the number of blue spots in the coating on the anode and the number of perforations of the anode. The quality of any particular coating is considered to be inversely proportional to the number of blue spots on the anode panel and directly proportional to the amount of coating remaining on the cathode.

The perforation-test cell was modified further, to evaluate the coating system after the coated tin-plate has been fabricated into closures. The cells are made with glass cylinders, specially prepared, with both ends bevelled so that closure can be forced on each end. The test procedure is the same as that for the original perforation test except that the time of electrolysis must be shortened to about two hours. By using fabricated closures, information is obtained concerning the fabrication characteristics of the coating.

Summary

A new testing technique has been developed for evaluating sanitary linings on tin-plate closures. This technique is based upon the theory that corrosion is an electrochemical reaction involving anodic and cathodic areas. The test-cell is described. By means of this cell, the anodic and cathodic areas are isolated so that the effect of both the anodic and cathodic reactions upon an organic coating can be studied. Experiments have shown that the effect of cathodic reaction upon the organic coating markedly controls the rate of corrosion at the anode. If the bond between the coating and the metal surface is deteriorated by the alkaline products of the cathode reaction so that the coating is loosened at the cathode, the rate of corrosion at the anode is increased. The theory is proposed, therefore, that both anodic and cathodic areas can occur on coated metal surfaces and that perforation of coated closures results from galvanic action. Previous investigations *(1, 2, 4, 5, 6)* have been concerned with what occurs at either anodic or cathodic areas. This investigation has shown that the reactions at both areas are interrelated.

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The Survival of Yeasts in Orange Beverage Base

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The survival times of certain yeasts in orange beverage base are presented. The results indicate that orange beverage base probably is not important as a source of yeast contamination in the finished beverage.

A common cause of spoilage in carbonated beverages is fermentation by yeasts *(2)*. Such yeasts may enter the beverage through improperly cleaned bottling equipment or in some cases, they may be present in the ingredients from which the beverage is prepared.

The present investigation was conducted in order to determine the probability of viable yeasts existing in orange beverage base at the time of its receipt by the bottler. The survival times of various yeasts in this product are reported.

Methods

The orange beverage base used in the following experiments was obtained from gallon containers prepared for commercial use. It contained the following ingredients: pasteurized orange juice concentrate, orange oil flavoring, orange coloring, citric acid, water and approximately one percent sodium benzoate. The pH was 3.28 and the Brix reading was 50.2. Preliminary plating on Sabouraud's dextrose agar using dilutions ranging from 1:10 to 1:10,000 disclosed no viable microorganisms. The beverage base used in all experiments was dispensed into sterile bottles in 99 ml. amounts.

The yeasts used in survival tests were: *Saccharomyces cerevisiae, Saccharomyces ellipsoideus,* five unidentified fermentative yeasts isolated from spoiled beverage samples (strains 3A, 12A, 14A, 24A, and 36A), and a nonfermentative *Rhodotorula* (strain 18) isolated from beverage.

Inocula were prepared by suspending the growth from three-day cultures on Sabouraud's dextrose agar in sterile distilled water. Suspensions of the various strains were adjusted to approximately equal turbidity.

Ascospore inocula were obtained by growing *S. cerevisiae* on carrot slices for one week and suspending the resulting growth in distilled water. Direct microscopic counts on these suspensions showed the ratio of ascospores to vegetative cells to be approximately 1:2.

A standard inoculum of one ml. was employed for introducing the suspensions into the 99 ml. bottles of beverage base. Immediately prior to inoculation, plate counts were made on the inoculum suspensions in order to estimate the number of cells introduced into each bottle.

The survival time of the yeasts in orange beverage base was determined at two different temperatures. One set of bottles was incubated at 30° C. and the other at 2° C. Bottles were pre-warmed or pre-cooled to their respective temperatures before inoculation. All the refrigerated bottles were kept in a constant temperature water bath during inoculation, plating and incubation. An air incubator was employed for the 30° bottles. Each experiment was run at least twice and the results averaged.

Each bottle was plated immediately (within one minute) after inoculation in dilutions of 1:10 to 1:10,000 using Sabouraud's agar. Dilutions below 1:10 were not made because of the viscosity of the medium and because of the inhibitory effect of the sodium benzoate. To guard against possible inhibition of colonies on plates of the 1:10 dilution by sodium benzoate, sufficient agar was added to give a final benzoate concentration of approximately 1/300 of one percent. In experiments in which low counts were expected, at least ten plates were made from the 1:10 dilution and the average count per ml. calculated.

Experimental Results

The first experiment was designed to test the survival of vegetative cells of the eight yeast strains in orange beverage base when stored at 2° and 30° C.

Plates were made immediately after inoculation and at successive 24 hour intervals for one week. Additional counts were made at two weeks and six weeks. As shown in Table 1, vegetative cells of yeasts inoculated

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TABLE 1

Percent Survival of Vegetative Yeast Cells in Orange Beverage Base

Strain	Cells Inoculated Per ml. of Base	30° C. Incubation — percent survival at:			2° C. Incubation — percent survival at:		
		<1 minute	1 day	14 days	<1 minute	1 day	14 days
Saccharomyces cerevisiae	360,000	0	0	0	43.6	0	0
Saccharomyces ellipsoideus	184,000	0	0	0	0.7	0	0
3A[a]	325,000	0	0	0	10.5	0	0
12A[a]	204,000	0	0	0	32.4	0	0
14A[a]	193,000	0	0	0	30.1	0	0
18 (Rhodotorula sp.)	1,640,000	0	0	0	10.5	0	0
24A[a]	210,000	0	0	0	68.1	0	0
36A[a]	180,000	0	0	0	71.7	0	0

[a] Unidentified fermentative strains isolated from spoiled beverage.

into base stored at 30° were killed within one minute. The average inoculum was 412,000 cells per ml. of beverage base. When the inoculated base was stored at 2°, growth was obtained for each strain on plates prepared within one minute after inoculation. The survival ranged from 0.7 percent for *S. ellipsoideus* to 71.7 percent for strain 36A. No organisms were detected on plates made at 24 hours or subsequently.

Since vegetative yeast cells incubated in the base at 2° survived an exposure of less than one minute but were dead within 24 hours, a portion of the experiment was repeated at 2° with more frequent plating. Suspensions of *S. cerevisiae* and *S. ellipsoideus* were employed as inocula. Counts were made at the time of inoculation and at fifteen minute intervals thereafter.

In agreement with the previous data, *S. ellipsoideus* was found to be more sensitive than *S. cerevisiae*. An inoculum of 470,000 *S. ellipsoideus* vegetative cells per ml. of base showed only a 0.7 percent survival within one minute after inoculation (Table 2). No growth was

TABLE 2

Percent Survival of S. cerevisiae and S. ellipsoideus Vegetative Cells at 2° C. in Orange Beverage Base

Strain	Cells Inoculated Per ml. of Base	Percent Survival At:					
		< 1 min.	15 min.	30 min.	1 hour	2 hours	1 week
Saccharomyces *cerevisiae*	618,000	23.2	6.1	1.2	0.2	0.0	0.0
Saccharomyces *ellipsoideus*	470,000	0.7	0.0	0.0	0.0	0.0	0.0

obtained on plates made after this time. *S. cerevisiae* survived somewhat longer. An inoculum of 618,000 vegetative cells per ml. of base gave a 23.2 percent survival on plates made within one minute and 0.2 percent at one hour. There was no survival beyond two hours.

The data presented thus far seem to indicate that vegetative cells of the yeasts employed do not survive in orange beverage base sufficiently long to be important in the spoilage of the final product. The next experiment was conducted to determine whether or not the more resistant ascospores might survive for a significant length of time.

The procedure followed was the same as employed for the previous experiments except that the inoculum suspension contained approximately 110,000 ascospores and 230,000 vegetative cells of *S. cerevisiae* per ml. Plates were made within one minute after inoculation, then hourly for six hours, then daily for seven days, and finally at weekly intervals up to five weeks.

As shown in Table 3, the bottles which were stored at 30° C. showed only a 1.0 percent survival within one minute after inoculation. No growth was detected on subsequent platings. A much longer survival was obtained for ascospores stored in the refrigerated base. There was a 13.6 percent survival of ascospores after one day of storage at 2°, and 0.2 percent of the ascospores were still viable at the end of one week. Evidence of complete sterility was not obtained until the third week of storage.

TABLE 3

Percent Survival of S. cerevisiae Ascospores[b] *at 2° C. and 30° C. in Orange Beverage Base*

Temperature of Incubation	Percent Survival At:				
	< 1 minute	1 day	3 days	1 week	3 weeks
2° C.	58.2[c]	13.6	3.2	0.2	0.0
30° C.	1.0	0.0	0.0	0.0	0.0

[b] The inocula employed in this experiment contained approximately 110,000 ascospores and 230,000 vegetative cells per ml. of base.

[c] This percentage was based on the total inoculum (i.e.: ascospores plus vegetative cells) since it was shown that vegetative cells of *S. cerevisiae* survive beyond one minute in flavor base stored at 2°. All other survival percentages in this table are based on the ascospore inoculum alone since it can be assumed from data presented in the other two tables that no vegetative cells have survived.

Discussion

When yeast spoilage occurs in carbonated orange beverage, one of the possible sources of contamination is the orange beverage base from which the final product is prepared by the bottler. The results presented in this report indicate, however, that the beverage base probably is not important as a source of contamination owing to the short survival time of yeasts introduced experimentally into this product.

If the base were refrigerated up to the time of use, it is conceivable that yeast ascospores introduced during manufacture might survive up to two or three weeks. In actual practice, however, the base is manufactured and stored at temperatures close to 30° C., a temperature at which experimental inocula far in excess of most natural contamination failed to survive beyond one hour.

The lethal effect of the orange base upon yeasts appears to be largely due to the one percent concentration of sodium benzoate although the low pH (3.28) is also important since, as shown by Cruess and Richert *(1)*, the toxicity of sodium benzoate for yeasts increases as the pH becomes lower.

It is interesting to note that although sodium benzoate is usually considered to function as an antiseptic *(3)* the one percent concentration present in orange beverage base is apparently quite zymocidal.

Summary

Eight strains of yeasts were shown to survive less than one minute when inoculated into orange beverage base (pH 3.28) containing approximately one percent sodium benzoate at a temperature of 30° C. When inoculated into base pre-cooled to 2° C., the same yeasts showed varying percentages of survival within one minute after inoculation. There was no survival on plates made at 24 hours or subsequently.

Ascospore suspensions of *Saccharomyces cerevisiae* were killed within one hour after inoculation into base held at 30° C., but similar suspensions survived up to two weeks in base maintained at 2° C.

Yeasts and yeast ascospores do not appear to survive sufficiently long in orange beverage base to be a source of contamination in the finished beverage.

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Foam Fractionation Procedures in the Isolation of Fruit Proteins[a]

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The application of foam fractionation procedures was satisfactory for fractionating the protein complex from extracts of apple tissue. Such procedures may provide practical methods not only for isolation of traces of material in solution or suspension, but also for purification of certain mixtures in which undesirable substances are capillary active or may be made capillary active.

While it has been recognized for some time that the components of certain solutions are adsorbed by foams which these solutions can form, few applications of this characteristic to concentration and fractionation techniques have been reported. The fractionation of mixtures by foams makes use of the fact that certain substances, being capillary active, have a tendency to concentrate at gas-liquid interfaces just as certain other substances are preferentially adsorbed at the liquid-liquid interfaces of emulsions. The phenomenon of capillarity as shown by the rising of water within a fine bore glass tube when the tube is dipped into water is well known. The double film which makes up the walls of a bubble may be considered as exerting an effect similar to that of the walls of the glass tube. Bubbles on the surface of a solution containing protein may take up the protein through capillary action and thus remove it from the solution by physical means. If a solution contains a mixture of proteins or other capillary active substances they may be fractionated from each other by dispersing a large number of bubbles throughout the solution. The materials having the greatest capillary activity will be removed first.

Since most proteins are capillary active, if a solution containing protein is frothed by means of a suitable gas disperser, a foam rich in adsorbed protein is produced. Frothing materially increases the surface of the system, and protein concentrates at the gas-liquid interface as the bubbles rise. The adsorbed protein may become irreversibly flocculated owing to its high concentration and surface denaturation occurring in the course of the process. Even under these conditions, however, the resultant material is well suited for studies on its composition, and the determination of certain of its characteristics.

The present report concerns the application of foam fractionation procedures to studies on the composition and nature of the protein complex of apple tissue. The technique provided a highly satisfactory method for concentration of the very small amount of protein complex present in extracts of the tissue which, because of the peculiar nature of the complex, would have been difficult to separate.

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Review of Literature

Ostwald and Siehr *(3, 4, 5)*, in studies of foam characteristics, conducted experiments on the concentration and separation of capillary active substances both colloidally and molecularly dissolved. Employing foam fractionation techniques, these workers reported the complete separation of albumin from potato and sugar beet juices, and described apparatus and methods for analysis by foam separations. The method was later applied to the quantitative separation of vegetable albumin from the raw potato juice, or "sweet water," remaining after commercial preparation of starch.

In an excellent review and critical discussion of recent papers, Shedlovsky *(7)* considered some of the factors involved in fractionation of mixtures by foams. Examples of the extent of adsorption at both liquid-liquid and air-liquid interfaces were cited from the literature, and various procedures were described. The discussion included the relationship of such factors as selected adsorption, composition and concentration of solution, pH of solution, temperature, size of bubbles, rate of foam formation, eddy currents in homogeneous foams, and the effects of bulk and surface viscosity. Among the investigations cited were some by Abribat *(1)* who asserted that drying of foams by drainage is responsible for the enrichment of the foam with respect to certain constituents in the solution, and the separation of numerous constituents is primarily a function of the difference in the flow characteristics at the gas-liquid interfaces and of the bulk liquid. He further claimed that this separation does not necessarily correspond to the surface activity of the constituents involved. With proteins, he maintained that the rigidity of the interfacial films of the foams is a function of both pH and temperature, and other factors being equal, the most suitable conditions for foam fractionation occur at the isoelectric point of the protein in question. However, the temperature should not exceed that value above which some proteins do not form viscous films and foams. While Abribat *(1, 2)* stressed the importance of differences in foam drainage, Schütz *(6)* designed an apparatus in which the foam was removed as soon as possible after formation, so that only a short time was allowed for drainage. He claimed that such a design had the advantage of foam automatically ceasing to be carried over if its stability suddenly decreased or increased when the surface-active material collected from the foam had all been removed from the solution. At this stage, the receiver could be changed, the gas pressure increased, and the next fraction collected.

Experimental

Apple tissue contains approximately 0.2 percent protein, and owing to the nature of the complex involved, procedures which effected satisfactory extraction of the complex called for large volumes of extractant. Such procedures likewise dissolved large quantities of

other substances, some of which, like pectin, were non-dialyzable and tended to follow the protein complex in and out of solution when common precipitation methods were employed. Removal of these substances by chemical means could not be accomplished without some degradation of the protein. Foam fractionation of extracts, however, resulted in preparations which were practically free from impurities carried along by the usual iso-electric precipitation methods or by flocculation with alcohol or acetone.

Observation of Foam Formation

Experimental studies under varying conditions and employing various solvents revealed that extraction of frozen apple tissue with 1.7 percent (0.05 M) sodium tetraborate solution by maceration in a Waring Blendor, permitted recovery in filtered extracts of 86 percent of the alcohol-insoluble nitrogen. Borate solution in an amount of 500 ml. per 50 gm. of frozen apple tissue was used for each charge of the Blendors. Observation of the formation of a stable foam in the course of the extraction procedure, and similar observations of foam formation in extracts during concentration procedures prior to employment of iso-electric precipitation techniques, ultimately suggested the possibility of foam fractionation of extracts as a means of separating the protein from the dispersed phase. Under the conditions of relatively large scale vacuum evaporation of extracts involved, the necessity for admitting air through a capillary to prevent violent ebullition resulted in the almost immediate formation of a stable foam which later condensed to a scum and precipitate of protein-rich material. It became apparent, therefore, that capillary active substances of a protein nature were involved in the formation of the foam, and that these substances might be fractionated from the extracts by frothing.

Apparatus

Preliminary investigations of foam characteristics were conducted with filtered extracts of apple tissue in columns of various diameters and heights, and under different conditions of gas flow. Compressed air was found to be suitable as a gas for the purposes of the present study. Data obtained from these investigations provided approximate dimensions required for an apparatus which would permit recovery of a reasonable amount of protein material from each charge of the apparatus. A finished apparatus modeled after the one designed by Ostwald and Sieher *(4)* with certain modifications, was constructed as shown in Figure 1. The column, 90 cm. in length, was made from 50 mm. Pyrex tubing, tapered at one end to a diameter of 12 mm. This end was bent to form an arc of 180 degrees, and to it was joined a section of 12 mm. Pyrex tubing to provide a suitable outlet for foam collection. A filter funnel, with fritted disc of very fine porosity 40 mm. in diameter, was joined at the opposite end as a gas disperser. A gas dispersing unit of this size possessed the advantage of permitting constant disperson of gas across the entire area of the column. The finer the dispersion of bubbles which could be effected, the more efficient the foaming process was found to be. The stem of the funnel

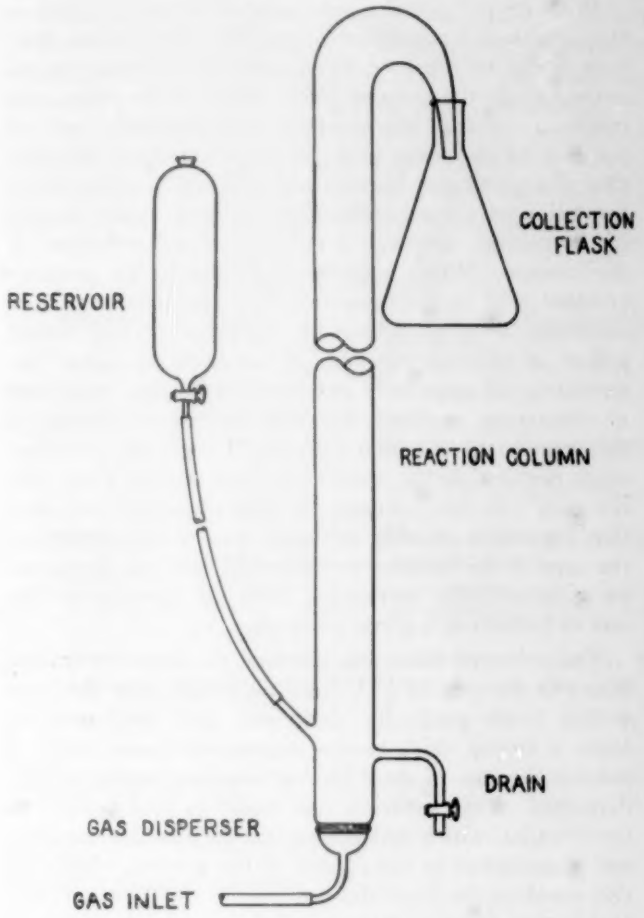

FIG. 1. Apparatus for foam fractionation.

provided a suitable gas inlet, and pressure of the gas at the disc prevented passage of any liquid into the stem. A cylindrical separatory funnel of 1500 ml. capacity served the dual purpose of a reservoir and a means of controlling flow of extract into the apparatus. A drain for removal of exhausted solution provided a system which made possible a semi-continuous process.

Procedure

In use, extract was fed into the column from the reservoir, and the presure and flow of gas was so adjusted that a well-drained foam issued from the outlet in a continuous stream. The foam collected was of such stability as to necessitate its collection in large containers, and Erlenmeyer flasks of 4-liter capacity were found to be highly satisfactory for this purpose. Approximately 1000 ml. of extract could be handled as an individual charge in this apparatus, and preparations of protein material were generally made from composite collections from twelve such charges of the apparatus. Experiments with borate extracts revealed the formation of three distinct foam fractions during the course of the process, successive fractions being revealed by noticeable changes in structure of the micelles. Extracts used in the present study were at pH values of 8.6 to 8.7; and under the conditions of the study, changes in pH of extracts above the approximate iso-electric range of pH 3.9 to pH 2.9 had little effect on foam formation.

With proper initial adjustment of height of liquid in the column and regulation of gas flow, the various fractions could be removed by incremental increases in gas pressure. In the present study, however, no effort was made to separate the fractions quantitatively, and all portions of the foam were collected together. Exhaustion of a particular fraction was denoted by cessation of formation of a foam sufficiently stable to reach the top of the column, although a stable foam still remained in the column. When a further increase in air pressure resulted only in the formation of a discontinuous foam structure, with an attendant discharge of free liquid, inflow of air was cut off. What froth remained immediately disappeared, indicating that the maximum of substances capillary active under the conditions of the experiment had been exhausted. With the low pressures required in the apparatus, little trouble with eddy currents was encountered. It was obesrved, however, that formation of eddy currents was closely related to the size of the bubbles produced by the gas disperser, the susceptibility increasing with an increase in the size of bubbles at a given pressure.

The collected foam was allowed to stand for several hours in the cold at 5° C., during which time the light yellow froth gradually darkened, and condensed to leave a heavy, dark-brown suspension from which a notable amount of dark-brown, powdery sediment was deposited. This sediment was found to be a portion of the complex which had undergone surface denaturation and coagulation in the course of the process. Material recovered in the foam did not lose its capillary activity, and in that form, or resuspended in fresh quantities of buffer solution or water, could again be frothed. Even the powdery sediment retained its surface activity, and was readily carried in the foam.

Repeated frothing in additional buffer or water suggested itself as a possible step in further purification of the material, but no increase in nitrogen content of collected material was observed if foaming of the original extract had been carried out under properly regulated conditions. Under these circumstances, a clear, colorless solution remained behind when frothing was completed. When extracts had been made from starchy apples, however, and relatively large quantities of finely divided starch had been carried over in the foam, resuspension in buffer or water and repeated foaming facilitated separation of the starch, which remained behind. This process had the disadvantage of being time-consuming, and when studies were made with starchy apples, starch was removed from the preparations by hydrolysis.

For studies on the composition of the complex, foam residues were flocculated by treatment with an equal volume of 95 percent alcohol acidified to a concentration of 1 percent hydrochloric acid, and the flocculated materia was separated either by centrifuging or by filtration through a fine hardened filter paper in a glass Buechner-type filter funnel. Flocculation in acidified alcohol possessed the advantage of effecting complete precipitation of the complex along with simultaneous removal of small amounts of alcohol-soluble impurities which were carried along with the protein. These impurities appeared to be for the most part closely associated flavones. Washed free of chlorides with water, followed by two additional washings with neutral 95 percent alcohol and one washing with absolute ether, the material dried rapidly in the air. Final drying was carried out under a vacuum of 29 inches at 65° C. for one hour. Dried preparations were light-brown in color, and in the form of a very finely divided powder. Although finished preparations were undoubtedly denatured when treated by this procedure, the material was in a form well suited for ultimate chemical and amino acid analysis.

Amino acid analysis of hydrolysates of the preparations indicated that such preparations were representative of the alcohol-insoluble nitrogen fraction of the apple tissue. A detailed account of these analyses, along with further studies of the ultimate chemical composition of the complex and studies on the nature of the protein systems, will be reported in a later paper.

Discussion

Protein preparations obtained either by iso-electric precipitation methods or by foaming appeared to be a complex of a number of protein systems in combination with a constant proportion of non-nitrogenous material which could not be separated without some degradation of the nitrogenous portion. This material was deep red-brown in color and relatively inert chemically, and appeared to be the agent which imparted the color to the preparations. Its composition and general properties, as shown by analyses, indicated that it was a phlobaphene or similar tannin-like compound. It was believed that the formation of a stable, water-insoluble, protein-tannin complex, very likely produced immediately upon rupture of the cells, may be responsible for unusual solubility properties attributed to the protein.

Such a complex dispersed in the extracts used offers interesting possibilities in regard to the mechanism involved in its separation from solution. A complex of protein and tannin, as considered from its application in the clarification of fruit juices, is known to coagulate and precipitate from solution, and in that form may represent a coagulated complex of the protein. If this assumption is correct, the capillary activity of the material in the present study may have been dependent on certain groupings in the protein molecule since the isolated tannin material in itself possessed no capillary activity under the conditions of the study.

Of particular interest in this respect was the observation that extracts, which had originally been cloudy to an extent that rendered them practically opaque, were brilliantly clear after frothing was complete. This observation suggested that the cloudiness of the extracts may have resulted from the presence of these surface active constituents in suspension, or from other substances held in suspension through the action of these constituents. Such a relationship in regard to protein material would tend to confirm the hypothesis of Carpenter and Walsh *(2)* that since proteins have long been known to be stabilizers of suspensions, it might be presumed that in juices protein acts in this way with respect to pectin substances.

Following this assumption, the possibility of clarifica-

tion of apple juice by direct frothing was attempted, but with little success. Under these conditions, a stable foam persisted only until the traces of capillary active substances present had been removed. These appeared as an actual coagulum in the foam, but no appreciable clarification of the juice could be observed following their removal. After standing in the cold, however, it was observed that suspended material settled out leaving a clear juice, again indicating the possible role of proteins in the stabilization of these suspensions. It is obvious that only very minute amounts are required to effect stabilization. Since no pectin is carried along by this procedure, foaming may offer a practical method for removal of traces of protein encountered in the preparation of pectin.

In cases where it is desired to minimize oxidation effects it is suggested that an inert gas such as nitrogen be used instead of air, as the "flotation" medium.

Summary

Application of foam fractionation procedures provided a highly satisfactory method for fractionating the protein complex from extracts of apple tissue. Analysis of the complex showed it to consist of a number of protein systems closely combined with a phlobaphene or similar tannin-like compound. The complex possessed certain solubility characteristics which indicated that the capillary activity of the material may be dependent on particular groupings in the protein molecule. Foam fractionation procedures may provide highly practical methods not only for isolation of traces of material in solution or suspension, but also for purification of certain mixtures in which undesirable substances are capillary active or may be made capillary active.

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An Investigation of a Mayonnaise Spoilage

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Bacillus subtilis and a yeast were isolated from spoiled mayonnaise. A survey of the production plant revealed sufficient unsanitary conditions to warrant the conclusion that gross contamination was the contributing factor to the spoilage of product.

The following investigation was started at the request of a mayonnaise manufacturer in the summer of 1947. The company which packed the mayonnaise involved in this investigation began the manufacture of mayonnaise and salad dressing on a small scale before the war and had expanded production considerably during the war years. In addition to the above mentioned products, pickles and pickle relish were also produced. All operations were carried out in a single plant and there was a large amount of sharing of equipment for the manufacture of the various products. The spoilage of mayonnaise became acute early in 1947 when the number of returned jars of short shelf life started to mount. Up to this time there had been no spoilage problem possibly because the undersupply during the war period insured a rapid removal of the product from the retail store shelves.

Mayonnaise is considered to be fairly resistant to microbiological decomposition because of its low water content and low pH. The few reports found on mayonnaise decomposition and spoilage of associated products have all listed some member of the genus *Bacillus* as the causative agent [*Bacillus petasites (2); Bacillus lekitosis (7); Bacillus vulgatus (4)*]. In view of the fact that the descriptions of the organisms involved in the cases of mayonnaise spoilage cited were not sufficiently complete for identification of the organisms, and because of the paucity of information on the subject, it was felt that the present findings are worthy of presentation.

Examination of the Product

The spoiled jars showed a slight separation of the product and were characterized by an "off" or slightly "yeasty" odor. Direct microscopic observations on both stained and unstained material revealed numerous spore forming rods (gram positive) and yeast cells. The organisms were distributed unevenly throughout the jars; individual samples from the same jar varied considerably in the number and proportion of organisms present. Samples of the spoiled product were plated on nutrient agar and Sabouraud's agar and incubated at room temperature for 48 hours. On many of the samples the results indicated as many as 1,000,000 organisms per gram. Gram stains of material from the various colonies, of which two types were evident, showed either gram positive spore forming rods or yeast cells. Several of the typical colonies were transferred to slants for further study and identification.

Identification of the Organisms

The identification of the aerobic spore formers isolated from the mayonnaise was made using the methods and keys proposed by Smith *(5)*. The four isolates obtained

from the mayonnaise sporulated on glucose agar within 48 hours at 28° C. The protoplasm of an 18 hour culture stained evenly with a weak methylene blue solution. The diameter of the rods, measured with a filar micrometer, was less than 0.9 microns. The spores, stained with 5% malachite green, were shown to occur centrally to terminally and the sporangium was not swollen. These characteristics are similar to those of the organisms contained in Smith's first group. A determination of various fermentative and biochemical properties gave the following results for the four isolates tested. Glucose, arabinose, and xylose were fermented with the production of acid when the organisms were grown on a mineral salts-sugar medium. After 48 hours incubation at room temperature on a medium consisting of 7 gm of proteose-peptone, 5 gm of glucose, 5 gm of NaCl, and 1,000 ml of distilled water the presence of acetmethylcarbinol was demonstrated by the formation of a red color after adding 0.2 ml 40% KOH and 0.6 ml 5% alpha naphthol (alcoholic solution) to 1 ml of the media *(1)*. Growth occurred when the organisms were inoculated on nutrient agar adjusted to a pH of 6. Both starch and gelatin were hydrolyzed. Nitrites were produced from nitrates. The formation of a black pigment was not observed when the organisms were grown on nutrient agar containing 0.5% glucose. Similar results were obtained when nutrient agar containing 0.05% tyrosine was inoculated. On the basis of the above reactions the four isolates were identified as strains of *Bacillus subtilis*.

The yeast isolates were grown on Lindegren's *(3)* grape juice agar and allowed to sporulate on gypsum blocks. The cells were almost uniformly round although a small number of oval cells were present. Ascospores were round and smooth, usually four per ascus. A sediment was produced in liquid media. On the basis of this information they were tentatively identified as a member of the genus *Saccharomyces (6)*.

The organisms isolated were heavily inoculated into jars of fresh, good quality, mayonnaise. After four months of incubation at room temperature the jars containing the yeast exhibited a slightly "yeasty" odor. There was no observable change in the jars inoculated with the bacillus. The slight or lack of alteration of the inoculated mayonnaise by these organisms may be due to a difference in composition or pH of the test mayonnaise, as compared to the product from which the organisms were isolated.

Examination of the Ingredients

In view of the high counts obtained in the spoiled product, and because of the resistant nature of the mayonnaise to bacterial spoilage, it was assumed that at some point in the production process gross contamination was occurring. Consequently an examination of the raw materials and a general sanitary survey of the plant was made. Bacterial populations were determined by plating the various ingredients on nutrient agar. A similar procedure using pasteurized ingredients (heated at 80° C. for 5 minutes) gave the spore count. Yeast and mold counts were made on Sabouraud's agar. Table 1 gives the results obtained.

TABLE 1

Plate counts on raw materials

Ingredient	Aerobic bacteria per gram [1]	Spore-formers per gram [1]	Yeast per gram [2]	Mold per gram [2]
Frozen egg	45,000	< 100	0	0
Sugar	2	0	0	0
Vinegar	0	0	0	0
Oil [3]	few	few	few	few
Starch	600	80	0	0
Garlic	30,000	4,000	500	2,000
Mustard	13,000	1,800	0	1,000

[1] Nutrient agar.
[2] Sabouraud's agar.
[3] No consistent count could be obtained because of emulsification difficulties. The numbers were very low.

The data show that only the egg and spice had appreciable bacterial populations and only the latter contained enough spore formers to be significant.

A general sanitary survey was undertaken of the mayonnaise production line by means of swab tests made immediately after the routine equipment cleanup at the end of a production day. Plant cleaning procedure consisted of pumping hot water through the homogenizer and pipe lines; this was followed with a hot 1% solution of lye which was removed with additional hot water. The equipment was not dissembled for cleaning. The innerface of the homogenizer vat averaged fewer than 100 organisms per square inch, and in all probability was not a point of gross contamination. The bacterial counts on the pipe inlet and pipe outlet from the homogenizer were higher, showing approximately 1,000 organisms as determined by swabbing one-half inch of the pipe. The interior of the pipe could not be reached and since this was not dissembled for cleaning it might have been a real danger point in operations.

The centrifugal pump on the homogenizer was dissembled and was found to contain fragments of pickle remaining from the manufacture of relish, which could serve as a source of yeasts and bacilli that would seed the product in the run of the next morning. The vanes of the homogenizer pump were not clean and bacterial counts varied from 1,000 organisms on a swab from a clean vane to almost 100,000 organisms on a swab from a vane which showed accumulated residues which had probably been left from previous runs.

The filling machine exhibited a number of potential danger points having two dead ends (previously inlet piping) in each of which residues had accumulated. Count from these areas were too high to interpret in terms of square inch of surface. The gasket by which the top portion of the filler was retained was also a bad source of contamination as it retained dried mayonnaise and relish from previous runs. More than 1,000,000 organisms were found on a swab streaked across the gasket joint.

Rinse tests of several containers showed a great variation in number of microorganisms varying from 170 to over 1,000 per jar.

Many other factors that could have contributed to the pollution of the product were observed. Some typical examples are:

1. The mayonnaise was conducted approximately 30

feet from the homogenizer to the filling machine by a continuous line that could not be dissembled for cleaning.

2. Lines conveying oil and vinegar to the homogenizer overhung it and the drip from the water condensing on the outside of these pipes fell into the homogenizer. Faucets over the homogenizer were not of the type that could be swung away from the machine when it was not in use.

3. Pumps were not dissembled routinely for cleaning.

4. Traffic from other sections of the plant, especially the pickle manufacturing section, passed by the homogenizer and filler. The dust stirred up could serve as another source of contamination.

5. Spices and sugars were stored in bags or open barrels and not in rodent, insect, and dust proof sealed containers or steel drums.

6. The homogenizer was used for the manufacture of pickle relishes and other products which provided contamination by yeasts and bacilli.

Summary

An aerobic spore former identified as *Bacillus subtilis* and a yeast, probably of the genus *Saccharomyces*, were isolated from spoiled mayonnaise. These organisms were present in large numbers in the product and probably were the cause of spoilage. A sanitary survey of the plant showed many unsanitary conditions and it may be concluded that the gross contamination of the product arising from these conditions was sufficient to cause spoilage even in such a microbiologically resistant product as mayonnaise.

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The Control of Household and Stored Food Insects by Contact Methods[a]

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REVIEW PAPER

A comparison of new and old insecticides, methods of application, and precautions in their use, is given.

It has been my experience that, although many will not admit it openly, every food handling and processing plant has a problem of insect control. With the first releases regarding the "miracle insecticide," DDT, many believed these troubles to be permanently ended. As yet such has not been the case, although these problems are, perhaps, more easy to solve today than 10 years ago.

It is common for the pest control operator to go into a plant and have the supervisor of one or more departments state that their section is insect free, and thus there is no need for pest control services. An experienced operator will tactfully suggest that perhaps he had better apply his materials as a means of preventing future infestations. This frequently discloses a heavy insect population. The unwillingness to admit the presence of vermin seems to be founded upon a belief that it is a personal reflection on the workers in a section to acknowledge their presence. This may often be true, to a certain extent, as uncleanliness and other unsanitary conditions are conducive to such infestations. The modern trained pest control operator will often make valuable suggestions as to the proper disposal of waste, or recommend structural changes which will serve to reduce or prevent insect growth, reproduction, and harborage.

In every plant there should be someone to whom is delegated the authority and responsibility to inspect the premises for vermin infestations. This person should be responsible only to the plant superintendent. At times, it is possible and desirable to employ someone for this from without the organization. This person will ordinarily make monthly or perhaps semi-monthly inspections and submit a written report on the conditions found. This frequent inspection is necessary due to the never ending introductions of new individuals of various species of insects. These are regularly brought in with shipments of many kinds. Also in congested areas, the source of the infestation may be a nearby plant or warehouse.

Although the experienced exterminator or sanitary inspector can quickly detect an insect infestation, it is the man who is daily on the job who often sees the first indications of trouble, and his reporting it to those responsible for its elimination will often enable the quick

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accomplishment of what might later become a much more difficult problem.

In general the whole concept of the control of household and stored food insects was much changed starting with the first use of DDT. Previous to its use, the pest control operator relied primarily upon space sprays, fumigants, and unstable or highly toxic powders. Were we today forced to depend solely upon those materials and techniques, we would most certainly feel handicapped.

A brief consideration of the various means of applying insecticides other than fumigants should be of value. First, let us consider space sprays, which are generally quick acting insecticides, that only kill those insects hit directly by the mist. The space sprays can further be divided into two types: (1) deodorized petroleum solutions designed for application by air pressure, and (2) aerosols which are most commonly propelled by compressed gases. The insecticidal aerosols now available, both gas propelled and heat generated, are primarily of value when used as space sprays and are extremely effective against many species of flying insects when an immediate kill is desired in a closed room. Repeated applications of aerosols directed into the hiding places of roaches and some other insect pests, although an expensive means of application, may be helpful in their control. Though gas propelled, the toxic ingredients contained in aerosol bombs are merely minute dispersions of solid or liquid particles, so the penetrating properties are not those exhibited by a gas, and therefore, are strictly limited. Aerosol application leaves little lasting effect and has virtually no value as a residual spray.

Dusts, another long used method of application, is in general a messy and unsightly manner of treatment. For use, dusts contain various percentages of the toxic ingredient ground in inert carriers. They are usually spread evenly over the surface being treated and are blown into all cracks and crevices. With limited equipment they may sometimes be more effectively introduced into inaccessible areas than liquid insecticides; however, the properly equipped operator with power driven sprayers and specialized nozzles can reach equally well every spot. The introduction of dusts, which are harmless to humans, to grain, nuts and other similar products before storage is a technique which offers considerable promise, but is at present not in general use.

As methods of application residual sprays have, only recently, had wide use in the control of household and stored food insects. The residual spray may be in the form of solutions, emulsions, or water dispersions. The selection of the form should be dependent upon the species being combatted, the toxic agent used, and the type of surface to which it is being applied. We rather proudly point back to our use of residual sprays as long as 10 years ago. The procedure used at that time, for the elimination of Buffalo moths, consisted of the application with a needle nozzle of a solution containing rotenone and other materials into areas frequented by the moths and yet where the toxic ingredients were not subject to decomposition by sunlight. In extreme cases, we very satisfactorily applied sodium fluoride in an emulsion form into cracks and crevices for residual protection from cockroaches.

It was only upon the wide use of DDT residual sprays that emulsions and wettable powders (long common in the agricultural and horticultural field) became generally accepted for use indoors. The economy, lack of fire hazard, and reduced danger to the operator through absorption by the skin, together with the fact that an oil solution should never be applied when it will fall on plants and vegetation, are all factors in favor of the use of water as a carrier. However, it is often desirable to use deodorized kerosene solutions containing other active ingredients along with the material desired for its residual properties. The wettable powder application, although probably the most economical, has the disadvantage of leaving a noticeable and sometimes objectionable powdery residue.

The toxic principles which I have found most desirable for use in pest control work are: pyrethrins, rotenone, DDT, chlordane and piperonyl butoxide.

Pyrethrum, a botanical, is an old stand-by that still fills an important place in the insecticide industry. When used as a space spray, it is very effective in producing rapid knockdown of a wide variety of flying and crawling insects. Also it has an irritating property which is helpful in locating areas of infestation. However, it has certain disadvantages. Insects receiving small amounts of pyrethrum may become paralyzed for a few minutes, or perhaps several hours, then completely recover. This recovery necessitated the sweeping up and burning of the flies killed by many old time insecticides. Pyrethrum undergoes rapid decomposition when exposed to light; this requires that it be stored in opaque containers unless protected by an efficient stabilizing agent.

Rotenone has largely been replaced for non-agricultural uses. Many consider the finest insecticides available seven or eight years ago to be pyrethrum-rotenone combinations. The pyrethrum supplied the rapid knockdown and then the slow deadly effect of rotenone assured a high kill. The relative lack of toxic hazards involved in the use of rotenone may cause us to again reconsider it for general use.

DDT is a material about which little need be said. It is slow in its lethal effect but minute quantities produce certain death to a wide variety of insects; however, it cannot be relied upon as the sole means of control of all species. Its effect on house flies and mosquitoes is truly amazing but satisfactory residual control of the common blow-flies, with the possible exception of the cluster-fly, has not been achieved with its use. It was quickly discovered that the 5.0% residual spray recommended for house-fly control was decidedly lacking in effectiveness against cockroaches. It was necessary to combine the use of DDT dusts and more concentrated DDT liquid sprays to eliminate the German roach by the use of DDT alone. However, the 5.0% residual spray is effective for killing a wide variety of moths and weevils when applied to the walls, ceiling, and all cracks and crevices. DDT should never be applied directly to any foodstuff nor should it be applied in a manner so that such products can become contaminated.

Chlordane (the technical material consisting of Octachloro-4, 7-methanotetrahydro-indane and related compounds) has caught the fancy of pest control operators, many of whom are primarily concerned with the cockroach. Like DDT, the first applications of chlordane were so successful that some again went so far as to predict that it would put the exterminator out of business, but with improved means of control the standards of efficiency expected have been raised until an inspector who previously would tolerate a moderate infestation now becomes highly upset over the presence of an individual insect. The refined chlordane is a heavy, viscous, high boiling liquid having a characteristic odor. It is completely soluble in deodorized petroleum distillates and in many common organic solvents, and as would be expected, is insoluble in water. It volatilizes slowly and when applied in the form of a dense fog is of some value as a local fumigant, though it is sufficiently non-volatile so that it is effective as a residue for several weeks. Again like DDT, it can be used as an oil solution, wettable powder, dust or aqueous emulsion. Two percent chlordane oil solutions and emulsions have recently become widely used in the form of a "stream spray" for treating small areas of infestation. This application consists of squirting the material by use of various types of dispensers into the cracks and crevices in which the insects hide. This method has the advantage of not contaminating nearby equipment or materials. The precautions required in the use of DDT are also necessary in the case of chlordane.

Piperonyl butoxide, (Butyl carbityl) (6-propyl piperonyl) ether and related compounds, is a material very unlike the others discussed. Alone, it is only moderately active in its toxic effect upon insects. But when combined with pyrethrins the synergistic effect is truly surprising, and in addition, it is reported to have a stabilizing property upon the pyrethrins themselves *(1)*. The toxicological data exhibited by the manufacturers is very complete and indicates that the combination of pyrethrum and piperonyl butoxide in various forms can be used with the complete lack of any toxic hazard to humans and other warm blooded animals *(1)*. This fact, together with its general effectiveness against a great variety of insects, including the housefly, cockroaches, all common weevils, moths and mites, is sure to cause it to receive wide consideration for use in all food handling and processing plants. It has proven very effective for combatting the cadelles, insects which have previously been difficult to control, and which are responsible for penetrating packaged products and thus leaving a means of entrance for other pests. Storage areas infested with weevils, moths, etc., have been treated with combinations of piperonyl butoxide and pyrethrum and have resisted further infestations for as long as several months. It was used experimentally last year in fly proofing and found effective for about six weeks in the control of housefly infestations.

Benzene hexachloride, a recently developed insecticide, is toxic in small amounts to a wide variety of insects. However, a characteristic unpleasant odor of the material currently available is an objection to its general use.

From the foregoing, it will be clear that no one insecticide component is the answer to all insect problems coming under the field covered by this paper. I have found it desirable to have available for use four deodorized kerosene type insecticides. One, a product designed for a special use in the tobacco industry contains only pyrethrins. A second product designed for freedom from toxic hazards to humans, together with a wide range of effectiveness, contains pyrethrins, piperonyl butoxide and rotenone. A third material which is effective against most any insect pest contains DDT, chlordane, piperonyl butoxide and pyrethrins. While a fourth contains only DDT.

The Department of Agriculture requires a caution statement on all insecticides containing DDT and chlordane. These statements should be read carefully. While the toxic symptoms of these materials on warm blooded animals are not quickly manifested, small quantities ingested over a period of time are accumulative and the effect will eventually become dangerous. Individuals within government control agencies have expressed the opinion that the detection of the presence of either of these materials, DDT and chlordane, renders a food unfit for human consumption and therefore, subject to seizure. Their opinions are receiving serious consideration. There is much toxicological data gathered on these materials and their accumulative effects, which, as yet, has not been released for publication. Its release should emphasize the desirability of using every precaution suggested.

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Heat Stability of Fat Peroxides[a]

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Extracted fat peroxides decomposed with increasing rapidity as the temperature was raised to 80°C. Extraction with boiling solvent yielded fat with lower peroxide value than obtained by Waring blendor extraction at room temperature. Development of peroxide appeared to be autocatalytic.

Organoleptic tests on frozen stored pork loins and sausage sometimes have indicated incipient rancidity before peroxides were found by chemical analysis in this laboratory. Fat for these analyses was extracted by the method devised by Conrad *(3)* for poultry skin. Ethylene chloride was distilled from the minced tissue until the water was removed and the fat was filtered off in solution in the solvent. The fat was protected from oxidation by displacement of air with solvent vapor, but no concern was felt for stability of peroxides which had already been formed in the fat.

However, Conrad and co-authors *(3)* suggested the possibility that during removal of the solvent in vacuo at 100° C. the fat peroxides may have been partially decomposed.

On the contrary, Ramsbottom et al. *(2)* found peroxide in fat after the solvent (chloroform) was evaporated (apparently in open air) from the fat. The fat was fresh pork fat containing no peroxide; it was extracted from fat tissue by a Waring blendor technique similar to the Watts *(4)* method, but without use of drying agent.

Procedure

Hot Versus Cold Extraction. When the Watts method appeared for fat extraction at room temperature, a series of sausage samples in various stages of oxidation was extracted in this Laboratory by both the Conrad and Watts methods. The peroxides in these two series of extracts are shown in Table 1.

In every case but No. 8 a distinctly higher peroxide was found in the extract prepared by the Watts method. No. 8 was freshly made from fresh pork and should have contained no peroxide. It seemed evident that peroxides were partially decomposed at the temperature of the boiling solvent (80° C.). The extent of decomposition probably depended upon the time required for extraction, which in turn depended upon the moisture content and degree of dispersion of the sample material.

Further evidence of the destructive effect of heat on fat peroxides is found in Table 2. Maximum values and good reproducibility were obtained by extraction with ethylene chloride at room temperature. Variable moderate oxidation appeared in samples rendered in air oven as compared with those rendered in vacuum oven at the same temperature. But more peroxide destruction than formation occurred at all three high-temperature methods as compared to the room-temperature method.

TABLE 1

Peroxide values found in a series of frozen stored sausage samples extracted with boiling ethylene chloride compared with those extracted at room temperature.

Sample No.	Peroxide Numbers[a] Boiling extraction	Cool extraction
1	11.0	16.1
2	0.2	3.7
3	0.2	3.1
4	13.2	22.1
5	0.0	0.3
6	0.0	0.1
7	0.2	2.8
8	0.0	0.0
9	0.0	7.4

[a] ml. 0.002 *N* thiosulfate per g. fat.

TABLE 2

Effect of extraction temperature and oven-rendering on peroxide values and their reproducibility. Duplicate portions for each procedure were taken from the same batch of moderately rancid sausage (well mixed)

Method of extraction	Peroxide duplication I	II
1. Ethylene chloride (boiling)	0.0	0.0
2. Ethylene chloride (room temperature)	7.4	7.4
3. Rendering (vacuum oven 100° C.)	1.9	2.0
4. Rendering (air oven 100° C.)	5.0	2.2

Heat Treatment of Fat Extracts. In order to test the heat stability of extracted fat peroxides, two lots of sausage in advanced stages of rancidity were extracted by the Watts method with ethylene chloride. The advantages of ethylene chloride over chloroform for this purpose are greater stability, lower volatility, and lower cost. The technical grade has never given a positive blank in use by the authors.

From the two sausage extracts 5-ml. aliquots were pipetted into a series of 20-cm. ignition tubes for peroxide determination by the Lea *(1)* method. The tubes were divided into groups for temperature-time tests as indicated in Table 3.

The 80-degree tubes were fitted to condensers and refluxed for the indicated time. Each peroxide value in the table represents an average of three replicates.

Although the authors have found peroxides practically unchanged in similar extracts after one week stored at 5° C., it is apparent that an increase of 13 percent occurred after five weeks for sausage A.

At 37° C. much more rapid changes occurred, and the trend of change seemed to depend upon the state of oxidation. Although there was an initial increase for both sausages over the initial peroxide content, there was a decrease at 48 and 72 hours for sausage A, but a continued increase for sausage B.

At 50° C. sausage A extract registered a 13 percent increase in peroxide during the first 24 hours and afterward only a negligible increase. At this stage the

[a] Contribution No. 382 from the Department of Chemistry, 159 from the Department of Animal Husbandry, and 148 from the Department of Home Economics, Kansas Agricultural Experiment Station.

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TABLE 3

Effect on peroxides of time that fat solution was subjected to varying degrees of temperature. Ethylene chloride extract of sausage prepared at room temperature

Temp. ° C.	Time hrs.	Peroxide numbers [a]	
		Sausage A	Sausage B
5	0	8.7	42.5
	35 [b]	9.8	.....
37	24	9.1	44.4
	48	8.4	53.3
	72	7.8	55.0
50	24	9.8	53.3
	48	10.0	59.7
	72	10.1	58.5
80	0.5	6.0	39.7
	1.0	4.1	27.8
	1.5	2.4	15.2

[a] ml. 0.002 *N* thiosulfate per g. fat.
[b] Days.

peroxide seemed to be forming at the same rate at which it was decomposing. Sausage B extract increased in peroxide 25 percent in the first 24 hours and rose another 15 percent in the next 24 hours. Here it reached a maximum and decreased slightly in the next period.

Peroxide formation appeared to be autocatalytic; in the higher state of oxidation (sausage B) rate of formation exceeded rate of decomposition by a far greater margin than occurred in the lower state of oxidation (sausage A).

At 80° C. peroxide decomposition occurred at a rapid rate for both sausages and in all periods. Since this is the temperature at which extraction was made by the Conrad method, it is obvious why low results for peroxide were obtained by that method. Time required for such extraction was about one hour.

Chloroform as Solvent. In order to compare the behavior of ethylene chloride with that of chloroform toward extraction and stability of fat peroxides, duplicate portions of the same sausage were extracted with each of the two solvents by the Watts method. The peroxide values are shown in Table 4.

The difference in peroxide values obtained by the two solvents is slight and insignificant, although they were slightly higher in the ethylene chloride extract. Greater difference appeared after standing 20 days in a refrigerator at 5° C. A significant increase occurred in peroxide in the chloroform extracts, but only a slight increase in one of the ethylene chloride extracts appeared.

TABLE 4

Relative stability of fat peroxides dissolved in chloroform and in ethylene chloride. Extracts stored at 5° C. for 20 days.

Solvent	Peroxide numbers		
	Initial	Final	Change
			%
Chloroform	44.0	49.9	+13
Chloroform	42.1	46.3	+10
Ethylene chloride	45.1	45.0	0
Ethylene chloride	43.2	44.7	+3.5

This limited experience indicated no significant preference for either solvent as an extractant for fat peroxides. But it did indicate a slight preference for ethylene chloride for holding fat peroxides unchanged a considerable length of time.

Summary and Conclusions

Higher values for peroxide were found in extracts of rancid sausage made at room temperature than in extracts made with the boiling solvent (ethylene chloride).

Pronounced changes occurred in peroxide content of extracts subjected to temperatures of 37, 50, and 80° C. for varying periods of time.

Peroxide generation in the fat extract above room temperature appeared to be autocatalytic.

Fat peroxide seemed slightly more stable in ethylene chloride than in chloroform when stored at 5° C.

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A Conductometric Method for the Determination of Ash in Refined Lactose

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A conductometric method for the determination of ash in refined lactose has been developed that is satisfactory for an ash concentration range of 0.001% to 0.200%. The method possesses greater accuracy and is much faster than the standard muffle procedure.

Introduction

Although considerable work has been done concerning conductivity measurements as a method to determine ash in sugar, most of it has been in fields other than milk products. Snell *(6)* worked on maple products, Lunden *(2, 3)* and Todt *(7)* on beet products, and Zerban and Mull *(8)*, and Zerban and Sattler *(4, 9, 10)* on cane products. Since previous work [b] has revealed that the determination of ash in milk whey by a conductance method has many advantages over standard muffle procedures, it was decided to attempt the adaptation of the Zerban and Sattler *(5)* procedure to a method for the determination of soluble ash in refined lactose.

The development of this method was undertaken with three objectives in mind: (1) to provide a time saving and accurate measurement of ash in refined lactose; (2) to prove or disprove the accuracy of both the muffle and conductance methods within a certain range of ash content; and (3) to compare the conductance method with the muffle method as to accuracy and speed of obtaining a result.

Experimental

Before trying to compare the two ash methods, it was necessary to adapt the conductance method of Zerban and Sattler *(5)* so that it could be used for refined lactose.

Preliminary experiments demonstrated that a concentration of seven grams of sample dissolved in 100 ml. of conductivity water was satisfactory for conductance measurements.

Zerban and Sattler *(5)* developed a constant to be used when calculating the conductivity ash in sucrose products. This constant is obtained by dividing the muffle ash by the specific conductance and is called the C-ratio. When an average C-ratio is calculated for a series of samples, it may be used to calculate the conductometric ash value for unknowns originating from the same geographical source. The average C-ratio times the specific conductance of the unknown equals the percent ash of the unknown. Experiment proved that the C-ratio works just as well with lactose as with sucrose.

A total of 45 different lactose samples were collected from the plant process during a four-month period. These samples had an ash range of from 0.0013% to 0.6290% by the muffle method. All the samples were dried, ground, and placed in tightly stoppered tins, and muffle ash and conductance ash were determined on each sample.

Muffle ash is determined in the following manner: A sample of from 20-25 g. is weighed into a weighed silica dish and then placed in the muffle, leaving the door open until ignition is complete. The door to the muffle is then closed and the temperature adjusted to 650° C. At the end of 16 hours the sample is removed from the muffle, cooled, and weighed.

The specific conductance (1/specific resistance) on each sample was found by weighing exactly 7.0000 g. of sample into a steamed Mojonnier bottle and adding 100 ml. of freshly prepared conductivity water. The bottles were then placed in a constant temperature water bath at 20° C. ± 0.5° for one hour, and the sample stirred occasionally to facilitate solution. Resistance readings were taken by using a type RC-conductivity bridge and a cell with a constant of 0.1.

The C-ratios were calculated from the specific conductances and muffle ash values of twenty-two representative samples, and the average C-ratio was found to be 432 ± 35 (Table 1). The conductance ashes were calculated using the average C-ratio, and these are shown in Table 2 along with the specific conductances, muffle ashes, and the plus or minus deviations.

TABLE 1

Determination of Average C-Ratio [a]

Sample Number	Muffle Ash	Specific Conductance × 10^6	C-Ratio
	%	*mhos*	
1 C	0.0357	79.4	450
6 C	0.0256	54.8	467
8 C	0.1810	435.0	416
10 C	0.2420	526.0	460
12 C	0.3850	1081.0	356
13 C	0.1531	308.0	497
17 C	0.0438	112.0	391
18 C	0.1055	299.0	353
19 C	0.0806	160.0	504
23 C	0.1140	245.0	466
24 C	0.0716	143.0	501
26 C	0.0404	99.0	408
1 R	0.0079	15.4	513
5 R	0.0110	26.2	420
12 R	0.0068	15.9	428
13 R	0.0122	31.8	384
18 R	0.0104	24.4	426
19 R	0.0072	16.8	428
20 R	0.0063	17.2	366
21 R	0.0072	20.4	353
28 R	0.0066	15.9	415
30 R	0.0128	25.6	499
Average			432 ± 35

[a] C-Ratio is the ratio of % muffle ash to specific conductance.

So far, all comparisons between the muffle ash values and the conductometric ash values have been based on the assumption that the muffle ash values are correct. Nothing was known of the accuracy of the methods themselves, and therefore experiments were conducted to check the accuracies of the two methods.

To check the muffle ash method a series of lactose

[a] Present address: Permanente Metals Corp., Trentwood Works, Spokane, Washington.

[b] Unpublished work by the author.

TABLE 2

Comparison of Conductometric and Muffle Ash Values

Sample Number	Specific Conductance × 10^6	Ash Calculated	Ash Muffle	Deviation
	mhos	%	%	%
16 R	15.3	0.0066	0.0013	+ 0.0053
3 R	16.3	0.0070	0.0024	+ 0.0046
15 R	18.5	0.0080	0.0033	+ 0.0047
29 R	17.9	0.0077	0.0031	+ 0.0046
8 R	22.2	0.0096	0.0042	+ 0.0053
10 R	18.0	0.0078	0.0045	+ 0.0033
2 R	16.7	0.0072	0.0047	+ 0.0025
24 R	20.9	0.0090	0.0052	+ 0.0038
22 R	19.0	0.0082	0.0052	+ 0.0030
20 R	17.2	0.0074	0.0063	+ 0.0011
28 R	15.9	0.0069	0.0066	+ 0.0003
12 R	15.9	0.0069	0.0068	+ 0.0001
21 R	20.4	0.0088	0.0072	+ 0.0016
19 R	16.8	0.0073	0.0072	+ 0.0001
9 R	40.0	0.0173	0.0104	+ 0.0069
18 R	24.4	0.0105	0.0104	+ 0.0001
5 R	26.2	0.0113	0.0110	+ 0.0003
23 R	19.0	0.0082	0.0115	− 0.0033
13 R	31.8	0.0137	0.0122	+ 0.0015
30 R	25.6	0.0111	0.0128	− 0.0017
14 R	18.2	0.0079	0.0130	− 0.0051
26 R	22.7	0.0098	0.0143	− 0.0045
1 R	15.4	0.0067	0.0079	− 0.0012
6 C	54.8	0.0237	0.0256	− 0.0019
22 C	114.0	0.0492	0.0294	+ 0.0198
1 C	79.4	0.0343	0.0357	− 0.0014
26 C	99.0	0.0428	0.0404	+ 0.0024
17 C	112.0	0.0484	0.0438	+ 0.0046
24 C	143.0	0.0618	0.0716	− 0.0098
19 C	160.0	0.0691	0.0806	− 0.0115
23 C	245.0	0.1058	0.1140	− 0.0082
18 C	299.0	0.1292	0.1055	+ 0.0237
15 C	160.0	0.0691	0.1425	− 0.0734
13 C	308.0	0.1331	0.1531	− 0.0200
8 C	435.0	0.1879	0.1810	+ 0.0069
10 C	526.0	0.2272	0.2420	− 0.0148
Average Deviation				± 0.0072

TABLE 3

Muffle Ash Method

KCl added	Ash muffle	Wt. added (KCl loss)	Wt. subt. (blank)	Wt. Ash corrected	Salt actual	Ash corrected	Error
mg.	*mg.*	*mg.*	*mg.*	*mg.*	%	%	%
2.4	3.9	0.0	2.4	1.5	0.0096	0.0060	37
4.5	2.3	0.1	2.4	0.0	0.0180	0.0000	100
6.6	7.5	0.1	2.4	5.2	0.0264	0.0208	21
10.3	5.8	0.1	2.4	3.5	0.0412	0.0140	66
14.9	6.1	0.2	2.4	3.9	0.0596	0.0156	74
20.0	15.8	0.2	2.3	13.7	0.0799	0.0548	31
20.7	11.4	0.2	2.4	9.2	0.0827	0.0368	56
24.8	11.0	0.3	2.4	8.9	0.0991	0.0356	64
30.0	17.2	0.3	2.4	15.1	0.1199	0.0603	50
34.6	23.0	0.4	2.4	21.0	0.1382	0.0839	39
45.2	39.6	0.5	2.4	37.7	0.1805	0.1505	17
60.2	52.4	0.7	2.4	50.7	0.2402	0.2023	16
66.3	61.5	0.8	2.4	59.9	0.2646	0.2383	10
Average							45%

samples were weighed accurately and known amounts of potassium chloride added. These were then ashed at 650° C. for 16 hrs. Several samples of the same lactose were ashed without adding the potassium chloride, and the average of the values thus obtained was subtracted from each of the samples containing KCl. The potassium chloride contained negligible moisture, but a slight loss of weight upon ashing was discovered by running several blanks. The proper proportion of this loss was added to each sample in the experimental series. In this way each experimental ash value was corrected for loss of KCl due to volatilization and the ash already present in the lactose. The results of this series may be found in Table 3.

The accuracy of the conductometric method was determined by weighing small amounts of potassium chloride and enough lactose to make exactly 7.0000 g. Potassium chloride was used because it is a highly ionized salt that corresponds to the actual ionized salt content of our lactose, and it is this ionized content which is measured conductometrically. The percentages of KCl were exactly the same as those of the muffle ash experiment. To the 7.0000 g. sample was added exactly 100 ml. of conductivity water, and after the sample was completely dissolved and the solution brought to 20° C. in a water bath, a reading was taken. C-ratios were calculated for all readings using the following formula:

$$\frac{\%\ \text{ash} \times \text{reading}}{\text{cell constant}} = \text{C-ratio.}$$

The cell constant is 0.1. The known percentages of ash were used to calculate the C-ratios, not the muffle ash values. The average C-ratio, 694, was used to calculate the conductometric ash values by the formula % ash = C-ratio × specific conductance, and then the value obtained for the blank was subtracted from each of the other values. The results of this experiment may be found in Table 4.

It will be noted that a different C-ratio is obtained when KCl is added to lactose. This is expected since the actual composition of the sugar solution has been changed by this addition. Zerban and Sattler *(5)* have found considerable variation in the C-ratios of sucrose samples having varied chemical compositions and originating from different sources.

As a final confirmatory check on the conductometric method, thirty-six representative samples of refined sugar were collected from the plant process and both muffle and conductometric ash were determined on each. The mufflle ash values ranged from 0.0053 to 0.0451%.

TABLE 4

Conductometric Method

KCl added	Reading	Specific Conductance × 10^6	C-Ratio	Calc. Ash (C-ratio = 694)	Ash corrected	Ash actual	Error
mg.	*ohms*	*mhos*		%	%	%	%
0.7	3550	28.2	340	0.0196	0.0068	0.0096	29
1.3	3040	32.9	564	0.0228	0.0100	0.0180	44
1.8	2260	44.2	597	0.0307	0.0179	0.0264	32
2.9	1660	60.2	684	0.0418	0.0290	0.0412	30
0.0	5400	18.5	508	0.0128	0.0000	0.0000	....
4.2	1310	76.3	781	0.0529	0.0401	0.0596	33
4.9	1065	93.9	851	0.0652	0.0524	0.0799	34
6.9	860	116.3	852	0.0806	0.0678	0.0991	32
8.4	720	138.9	863	0.0964	0.0836	0.1199	30
12.6	500	200.0	902	0.1388	0.1260	0.1805	30
Average			694	Average			33

An average deviation between the values of the two methods was found to be ± 0.0063.

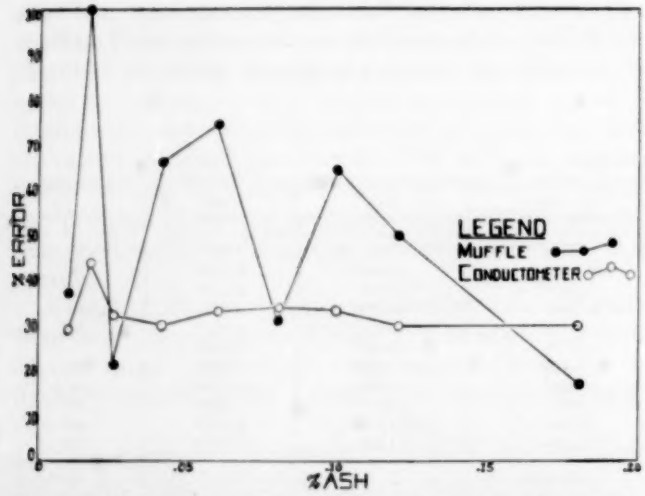
Discussion

For the proper interpretation of the results in this report it must be kept in mind that the muffle ash method is entirely empirical and yields discrepant results in the hands of different analysts. When the ash content is very small, as in many of the samples recorded in Table 2, the results of the muffle method are quite unreliable because only a few milligrams of ash are actually weighed. Unless very large quantities of sugar are ashed, which is precluded in actual practice, the possible percentage error in the ash figure becomes enormous. The measurement of specific conductance, however, is well known to be comparatively more precise, and for this reason alone the electrical ash method is to be preferred over the muffle ash method.

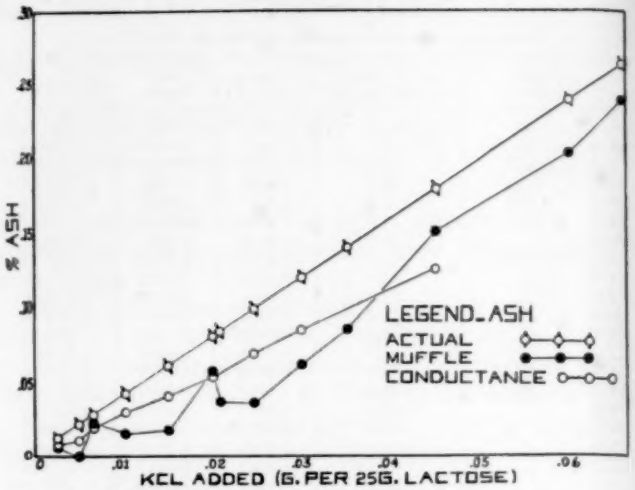
The deviation between the two ash methods is shown in Table 2, and the average deviation is shown to be ± 0.0072%. The results of sugars having muffle ash values higher than 0.24% were not included in the table since the deviation becomes greater and no practical correlation could be obtained. This is no criterion for concluding that the conductometric method for ash in sugars would not work satisfactorily for the higher ranges of ash content, since it is possible that by using less than seven grams sample per 100 ml. water for the resistance measurements a satisfactory procedure could be worked out. However, the method as outlined in this report is limited to ash percentages of 0.0010% to 0.2000% in lactose from the same geographical source and produced under uniform conditions.

In comparing the accuracies of the two methods it should be noted that the conductometric method is more accurate and consistent for amounts of ash lower than 0.15% (Graph 2). The consistency alone should prove its superiority, and when the percents of errors are compared, as in Graph 1, there can be little question. The average percent deviation from the theoretical amount of ash by the muffle method for ash amounts of less than 0.20% is 51% as compared to 33% for the conductometric method as performed in this laboratory.

One possible source of error in our accuracy check of the conductometric method is that the ratio of ash to



GRAPH 1. Comparison of the % errors of the two ash methods.



GRAPH 2. Comparison of the accuracies of the two ash methods.

sugar enters into the relation between ash content and conductivity of a sugar solution, and for a constant amount of sugar this ratio changes with the ash content *(1)*.

Conclusions and Summary

The electrical method for the determination of ash in lactose is satisfactory for an ash content up to 0.2000%, and is therefore especially suited to refined lactose. It is 18% more accurate on the average than the muffle procedure, and is much more rapid and easily executed. The contrasting times to obtain a result in this laboratory are as follows:

Muffle method	17 hours
Conductometric method	¾ hour

Acknowledgment

We would like to take this opportunity to thank Mr. J. R. Harrower for his advice and encouragement.

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Notes and Letters

By-Products of the Fishing Industry[a]

The principal types of fish landed at New England ports fall into two main categories, the fatty and the non-fatty varieties. The latter so-called "white" or "ground" fish varieties are made up of fish such as the cod, haddock, and pollack, and the former consists of fish such as the rosefish, mackerel, and herring. In the case of the rosefish, until approximately fifteen years ago no attempt was made to catch this fish commercially, the small amount landed being used almost entirely for bait. Today, however, it comprises the bulk of fish landed at Gloucester. The waste yield on the rosefish is approximately 70%. When one considers that over one hundred thousand tons of this variety of fish are landed at New England ports throughout the year, the volume of raw material for by-product manufacture can be appreciated.

In addition to the difference in the fat content of the fish landed, there is another very significant difference, particularly as it relates to the by-product industry, and this is the fact that the bulk of the white fish landed does not contain the viscera. These fish are eviscerated or "gutted" at sea and the visceral contents thrown overboard, with the possible exception of the livers, particularly in the case of codfish livers. Thus the first actual by-product of the fisheries is not available at the present time. Just how much value is being lost cannot readily be determined, but it is known that the visceral contents, including the glands and livers, contain valuable nutrients essential to poultry and animal nutrition.

There is still another loss of by-product material before the fishing boats land their catch and that is in the so-called "trash fish" brought up with each haul. These fish are either too small or not considered suitable because of variety, and are thrown back into the ocean. We understand that this "trash fish" constitutes an appreciable percentage of each catch. It has been said that throwing these fish, particularly the small ones, back into the ocean helps safeguard against depletion. We understand, however, that unfortunately these small fish are usually dead, either by suffocation or injury in the net, before being returned to the sea.

The principal by-product of the fishing industry is the waste fish itself, or that part of the fish remaining after the edible portion has been removed. It is customarily called "gurry" and consists mainly of the heads, backbone, skins, and tails, and, of course, the viscera, in the case of the fatty varieties. There is also considerable tissue attached to the bones, as only the fleshy portion of the fish is removed in the fillet. The gurry from the cutting tables is accumulated in hoppers or steel tanks at the filleting plants and is transported in tight-bodied trucks to the by-product plants for processing. In the past these by-product plants in general were extremely unpleasant and even obnoxious, with little or no effort being made toward control of spoilage or escaping odors. A visit to a modern plant today would reveal that good housekeeping practices and proper combustion control of non-condensable gases from the vapor condensing equipment have eliminated the unpleasant characteristics formerly considered inevitable with this type of operation.

The principal by-products manufactured from the waste fish are fish meal, fish oil, and condensed fish solubles. Of lesser importance, from the standpoint of volume, is the manufacture of fish glue and fish liver oils.

The value of the principal by-products lies in the field of animal and poultry nutrition. The fish meals are valuable for their protein, vitamin, and mineral content; the fish oils for their vitamin content and, to a lesser degree, their fat content; the condensed fish solubles for their vitamin content, particularly the several water soluble vitamins known to be present, and also for their protein content. Both the fish meai and the fish solubles contain the so-called "animal protein factor" so essential in poultry nutrition.

In general, there are three principal systems for the reduction of fish waste in use in New Enland, namely: the wet reduction process; the dry reduction process; and a wet digestion process.

The wet reduction process was designed essentially for the processing of the fatty varieties for the purpose of oil recovery. In this process, the fish waste is fed into a continuous cooker in direct contact with live steam. This cooks and disintegrates the fish in preparation for the pressing operation. The pressing is usually done in continuous horizontal screwtype presses. The press liquor containing the oil is run into settling tanks or is centrifuged to remove the oil. The press cake is dried in continuous driers, and the liquor from the oil separation, which is called "stickwater," is concentrated to 50% solids, usually in multiple effect evaporators. This liquor at one time was discarded, as it was considered uneconomical to attempt to concentrate it. However, because it proved to be a source of pollution of rivers and harbors, another means of disposal had to be found. This led to the development of a product known as "condensed fish solubles" which is prepared by treating the stickwater with acid and then concentrating it to approximately 50% solids. The product is marketed in drums or tank cars.

The dry reduction process consists of dehydrating either the fatty or non-fatty fish waste in steam-jacketed batch type driers, generally operating under a vacuum. In this process, the oil and the "fish solubles" are not separated but remain incorporated in the finished fish meal.

The wet digestion process for reducing fish waste is a patented process, and the details are not available. It is understood, however, that the oil is removed as in the

[a] Presented before Northeast Section, I. F. T., February 7, 1949.

wet process, and then the waste fish is treated so that there is a partial breakdown or hydrolysis of the protein, and the liquefied product is concentrated to approximately 50% solids for marketing.

The manufacture of glue from fish waste is confined almost entirely to codfish waste, in particular the skins, although the bones, trimmings, and heads are also used, when available. There is considerable demand for this glue in photo-engraving.

The manufacture of fish liver oils is limited to the supply of fish livers. Limited quantities of halibut, swordfish, and tuna livers are processed and nearly all of this oil is medicinal, the vitamin A content ranging from 50,000 to 300,000 units per gram.

The low potency liver oils such as the cod, dogfish, etc., are mixed with the fish body oils from the rosefish, sardine, etc., and utilized in feeding oils. These range in potency from 1,000 to 2,000 units per gram.

In general, the volume of liver oils produced is not great, as it is difficult to get the fishermen to save the livers.

The present value of fish oils lies in their vitamin A and D content; however, the recent development of a refining process for fish body oils in which the vitamins may be concentrated as much as thirty-five times and the residual oil fractionated to produce oil fractions suitable for use as drying oils for paints, low iodine value edible oil suitable for hydrogenation to margarine and cooking fats, and soap manufacture, will materially increase the value of these oils. Although at the present time the initial investment appears high, the process manufacturers indicate a high return.

Further possibilities of other by-products from fish waste seem unlimited. The preparation from fish waste of enzymes, hormones, protein hydrolysates, peptones, and numerous other products have been reported. For an insight into this vast potential, see "Possibilities in Developing Fisheries By-Products,"[b] a review presented by Dr. H. L. A. Tarr of The Fisheries Research Board of Canada at the 1947 Seventh Annual Food Conference in Boston, in which he outlines some of the work reported in the literature and which is being carried on at their experimental stations for by-product research. Studies along this same line, we understand, are being carried on at the Seattle Laboratory of the Fish and Wildlife Service.

In addition to the above products, large quantities of fish waste are canned or frozen for use in cat and dog, and fox and mink foods.

The studies now being made on the freezing of fish at sea and bringing the fish in round are being followed with great interest. If this proves feasible, the valuable visceral contents will be made available to the by-product industry. In view of present knowledge and investigational work in progress, it seems likely that there are great future possibilities for fishery by-products.

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[b] FOOD TECHNOLOGY, 2, 268 (1948).

Comparative Heat Penetration Studies on Jars and Cans[h]

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Introduction

The increase in the use of glass jars as containers for commercially canned foods during the war years has resulted in many requests for process information. All too few data were available on the heating characteristics of foods packed in glass, and most processes were based on those for similar products packed in metal containers of corresponding sizes, sometimes with a small safety factor added. This was an unsatisfactory situation, which was brought to a head in February, 1946, by a request from the California State Department of Public Health for processes for all commercially canned low-acid foods in glass containers of various sizes.

In September, 1946, a committee was formed to outline experiments for determining comparative rates of heat penetration in jars and cans of corresponding sizes. The object was twofold. First, to determine what modification, if any, of C. Olin Ball's *(2)* mathematical method of process calculation for cans was necessary to make it applicable to glass. Second, to determine, if any factor or factors could be developed which, when applied to processes for foods packed in metal containers, would give corresponding processes for similar foods packed in glass containers.

The project was a cooperative one, sponsored jointly by the National Canners Association, the Glass Container Manufacturers Institute, and the University of California Laboratory for Research in the Canning Industries, G. W. Hooper Foundation for Medical Research.

Original plans called for the study of convection, conduction, and "broken curve" heating rates in jars and cans of corresponding sizes. For the filling medium, it was decided to use aqueous bentonite suspensions *(4)*, one percent for convection heating, five percent for conduction heating, and an intermediate concentration for broken curves. Preliminary experiments with concentrations of 2.0 to 3.5 percent bentonite showed that it was not possible to prepare a suspension which would give a break in the heating curve at the same temperature on repeated heating. Results were so variable that attempts to compare "broken curve" heating rates were abandoned, and all subsequent heat penetration tests were made with one percent or five percent bentonite suspensions.

The following factors were studied with bentonite as the filling medium:

1. Convection and conduction heating rates in jars and cans.

2. Influence of position of jars in the retort on the heating rate. Three positions were investigated—cap up, cap down, and horizontal.

3. Influence of length of come-up time on the lethality of the process for one percent bentonite.

4. Influence of vacuum on the rate of heat penetration.

5. Lethality of the cooling period for one percent bentonite, and character of the cooling curves for five percent bentonite.

6. Comparison of rates of heat transfer in cans heated in water and in steam.

The heat penetration runs were divided into two series: preliminary and general. In the preliminary runs, studies were made of such factors as the character of the bentonite suspensions to be used; the stability of bentonite suspensions on repeated processing; type of thermocouple best adapted to the work; methods of introducing thermocouples into the jars; effect of vacuum on the rate of heat penetration; the lethality of the cooling period; determination of points of slowest heat penetration; and some work on the effect of jar position on the rate of heat penetration.

General runs included studies on the significance of jar position; comparison of heating rates in jars and cans of corresponding sizes; comparison between short and long come-up times for the effect upon rate of heating; and comparison between heating rates in jars and cans heated in steam and in water. Retort temperatures of 212° F., 240° F., and 250° F. were used, with short and long come-up times at 240° F. and 250° F. and a short come-up time at 212° F. The steam cooks were made only at 240° F. and 250° F. with short come-up times.

To supplement the work on bentonite, several inoculated experimental packs were made, using jars and cans of corresponding sizes. Products included were: strained pea soup; strained mixed vegetables with bacon; chopped mixed vegetables with chicken; peas in brine; and whole kernel corn in brine. Heat penetration determinations were also made on these products.

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Equipment and Procedure

Containers. The following sizes of jars and cans were studied:

Container	Dimensions	Weight of Jar (oz.)	Capacity (fl. oz.)	Fill of Bentonite Suspension at 150° F.	Headspace (Percent)
Baby Jar	202x309	3⅜	5.1	135 ml.	10.5
Junior Jar	208x401	4 15/16	8.1	220 ml.	8.5
No. 303 Jar	303x411	7	17.0	455 ml.	9.5
No. 2½ Jar	401x414	11	28.3	780 ml.	7.0
Baby Can	202x214		4.8	Full Can	
Junior Can	211x210		6.5	Full Can	
No. 2 Can	307x409		19.7	Full Can	
No. 2½ Can	401x411		28.6	Full Can	

No. 2 cans were included for comparison with No. 303 jars because No. 303 cans (303x406) were not available.

Bentonite. One percent bentonite suspensions were used to determine convection-heating rates, and five percent suspensions for conduction-heating rates. Stock suspensions of five percent bentonite were prepared as follows: The proper weight of bentonite (calculated on the dry basis) was added to 540 ml. of distilled water and thoroughly mixed in a Waring Blendor. The mixture was allowed to stand for 10 minutes and then decanted to eliminate sediment. The suspension was heated to 150° F. and canned in No. 2 cans (a considerable quantity was made at one time, and held for future use). After the cans were sealed, they were given two stabilizing cooks of about 2½ hours each at 250° F. The one percent bentonite suspensions were obtained from the stock five percent suspension by dilution.

Retort. The retort was vertical, 30 inches in diameter and 33 inches deep. The baffle plate was 7 inches above the bottom, and was perforated with 1-inch holes on 2½-inch to 3-inch centers. Steam was brought through a 1-inch line to a 1-inch air opening control valve, and thence through a ¾-inch line to a ¾-inch cross in the bottom of the retort. The holes in the cross were so located as to direct the flow of discharging steam horizontally to give a swirling motion to the water.

The bulb of the mercury thermometer was within the retort shell and about half-way between the baffle plate and the top of the retort. During the first runs, the bulb of the temperature controller was placed within the retort shell and between two legs of the steam cross. It was found that with the bulb in this position, there was a gradual increase in temperature during the process of 1° F. to 1.5° F., as indicated by the mercury thermometer. For all later runs, the bulb was situated within the retort shell and about one inch above the baffle plate. With this placement of the controller bulb, the temperature at the mercury thermometer remained constant during the process.

Compressed air was introduced into the steam cross through a valve connection close to the retort. During the water cooks, air was bubbled into the retort continuously to maintain circulation of the water.

Excess pressure was released at the top of the retort, through a Lunkenheimer pressure relief valve set at 30 pounds. A by-pass, containing a hand operated valve, was provided for releasing water during the cool. This 30-pound pressure was maintained at all processing temperatures.

A cooling ring of ½-inch perforated pipe was located just below the rim of the retort, and water entered this ring through a ¾-inch line. With this system, it was possible to reduce the temperature of the retort approximately 100° F. in 5 minutes, the rate of cooling adopted for all runs.

Temperature control was provided by a controller-recorder, with two dials, with one of which cams could be used to operate the control mechanism of the other. Cams were cut for temperatures of 212° F., 240° F., and 250° F. and for 5-minute and 20-minute come-up times. The 20-minute come-up time could be satisfactorily regulated in this manner, but it was found that the 5-minute come-up could not. Consequently, the shorter period was regulated by hand operation of the steam valve.

Thermocouple wires were passed out of the retort through a 1½-inch hole in the retort shell. Lead wires were wrapped with electrician's friction tape to make a plug large enough to seal the hole.

FIG. 1. Retort and Potentiometer.

Potentiometer. For determining temperatures in the cans and jars, an automatic indicating potentiometer was used, having connections for 24 thermocouples, selected by push buttons.

Thermocouples. Considerable time was spent in developing a thermocouple rod which would not leak even under high vacuum. The old method of using a plastic separator inside ¼-inch plastic tubing, with the ends sealed with some type of cement, was found to be unsatisfactory. Finally, in cooperation with Mr. Prentiss Cole, of the Cole Manufacturing Company, Palo Alto, Calif., a molded bakelite thermocouple was developed which was hermetic for all practical purposes. Copper-constantan couples of B. & S. No. 24-gauge wire were used, with glass fiber insulation. The bakelite rods were molded over the insulation. The thermocouple lead wires were continuous 24-gauge from the tip, since it was found that soldering lead wires to the thermocouple wires set up what appeared to be secondary couples on the constantan side. The wire used was obtained from Leeds and Northrup (1938 calibration) and was of the best thermocouple grade.

On standardization, no couple was found to be in error more than plus or minus ¼° F.

Methods of Closing Jars and Introducing Thermocouples

1. *Clamps.* Some couples passed through caps, which were fastened to the jars by means of clamps. In the photographs are pictures of the clamps in place, and of the retort assembly. A cut rubber gasket 3/32-inch thick was used between the rim of the jar and the cap.

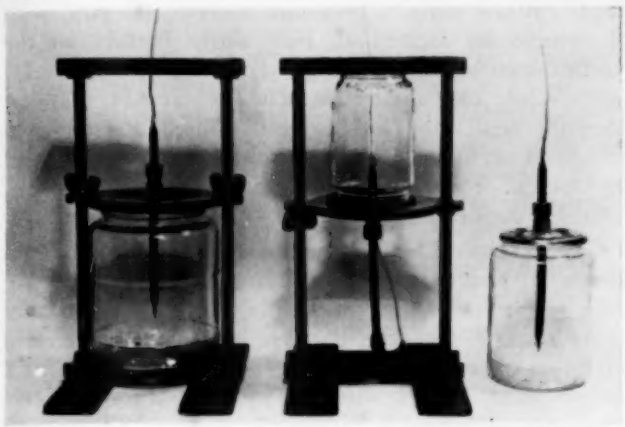

FIG. 2. Clamps and Thermocouples.

2. *Rubber Stoppers.* In certain runs, the thermocouple was introduced through the side of the jar. To accomplish this, a ½-inch hole was drilled through the side of the jar at the desired distance from the bottom, and the thermocouple was inserted through a rubber stopper of adequate size to seal the hole. The jar was closed either with a White cap or with a clamp. This method of introducing the thermocouple was not too satisfactory, owing to shrinkage of the rubber under the influence of heat. When this occurred, there was usually some shifting of the thermocouple position during the process, and a number of the jars leaked during the cool.

3. *Soldered Packing Glands.* A number of experiments were made with thermocouples introduced through packing glands soldered to the jar lids. This procedure was found to be satisfactory and was used with the Owens-Illinois "Hermeseal" rolled-on closures for all No. 303 jars in the general runs.

All three methods of inserting thermocouples are illustrated in the photographs.

Introduction and Location of Thermocouples in Cans. The same type of thermocouple was used for the cans as for the jars. Thermocouples were introduced through packing glands soldered to the sides. In cans containing one percent bentonite, the thermocouples were located ½ to ¾ inch above bottom, while the center position was used for five percent bentonite. All cans were processed in a vertical position.

Initial Temperature and Vacuum. A uniform initial temperature of 120-130° F. was maintained for the one percent suspensions, and 140-150° F. for the five percent suspensions. Vacuum was obtained by hot fill, about 150° F. for both cans and jars.

Frequency of Temperature Readings. Readings were taken at ½- or 1-minute intervals during rapid heating and 5-minute intervals during slow heating.

Number of Runs. In most runs, Junior and Baby containers were processed together, and No. 303 and No. 2½ together. When one lot of containers was set up in the retort, a series of either three runs or five runs was made—three runs if the medium was five percent bentonite (at 250° F., 240° F. and 212° F.) or five runs if the medium was one percent bentonite (two come-up times at 240° F. and at 250° F., and the short come-up at 212° F.). The assembly was then torn down, fresh bentonite and different thermocouples were used, and a duplicate series of runs was made.

Data Obtained

Data were evaluated in terms of lethalities by the general (graphical) method *(3)*, and heat penetration curves were plotted on semi-logarithmic paper. The following values were obtained on the general runs:

R.T. — retort temperature.

I.T. — initial temperature at the slowest heating point.

CUT — come-up time to retort temperature.

$m + g$ — difference in degrees between retort temperature and cooling water temperature.

f_h — the slope of the straight line portion of the semi-logarithmic heating curve expressed as minutes required for the curve to traverse one logarithmic cycle of temperature.

j — a factor which, when multiplied by the difference between the retort temperature and the initial temperature, designates the point of intersection of the vertical straight line representing the beginning of a process with the extension of the straight line portion of the semi-log heating curve, when no time is consumed in bringing the retort to processing temperature.

F of the total process at 240° F. and 250° F.

F symbolizes the lethal value of a process at the critical point in the container, expressed in terms of the number of minutes required to destroy an organism at 250° F. A process that is just capable of sterilizing a food containing bacterial spores which require F minutes heating at 250° F. for their destruction is said to have a lethal value of F minutes. The symbol F_0 is used when $z = 18$. z is the slope of the thermal death time curve on semi-log paper, and represents the number of degrees F. required for the curve to traverse one logarithmic cycle of time. The F value is used to compare the sterilizing values of different processes.

F_{212} of the total process at 212° F.

F_{212} symbolizes the number of minutes required to destroy an organism at 212° F. when $z = 18$.

F_o or F_{212} of the cool.

F_o or F_{212} of the process to the start of the cool (by difference). Process for one percent bentonite suspensions required to obtain an F_o or F_{212} of 8 (lethality of come-up time and cool included).

e — the lethal value of the come-up time, in terms of holding time at the retort temperature, expressed as percentage of come-up time.

With the one percent suspensions, the significance of comparative results was frequently obscured by fluctuations in retort temperatures and variations in the earlier thermocouples used. Corrections, therefore, were made on the lethality curves, continuing the curve to lethality at 212° F., 240° F., or 250° F. if the couple attained constant readings below this value, or reducing the lethality to that at retort temperature if the thermocouple attained constant readings above this point. A slight variation in temperature near the retort temperature, particularly at 250° F., produces a far greater effect on the lethality of the process than a considerable variation at a lower temperature. Most of the temperature variations requiring correction were 1° F. or less, and the corrections were felt to be justifiable.

Preliminary Runs

Heating Characteristics and Stability of Bentonite Suspensions. One percent, five percent, and intermediate concentrations of bentonite suspensions were studied. It was found that one percent was satisfactory for convection heating and five percent was satisfactory for conduction heating. No intermediate concentration used (from 2.0 to 3.5 percent) would give consistent results with broken curves. With each run on the same suspension, the break occurred at a different time and, after only a few runs, the heating curve became a straight line. Consequently, plans for using bentonite to establish comparative heating rates for broken heating curves were abandoned.

Following two stabilizing processes, suspensions of five percent bentonite in No. 303 jars were processed 14 times at 250° F. for 75 to 100 minutes to determine whether any change occurred in the character of the suspension which might result in a change in the rate of heat penetration. Two jars were left undisturbed and two others were shaken vigorously between runs. No significant difference in f_h was observed for the first 10 runs in either the agitated or non-agitated jars, but in subsequent runs the f_h values were not consistent. The number of times any suspension of five percent bentonite was heated in the general runs never reached 10.

Four runs were made to check the stability of one percent bentonite in No. 303 jars. These jars were processed with others containing five percent bentonite, and so received excessively severe processes. No significant change in f_h values was noted in any run. Further runs were considered unnecessary, because the one percent suspensions were not used more than 5 times, with comparatively short processes.

Experiments were made to determine the effect on the rate of heat penetration of variations in the concentration of bentonite. Runs were made with the following concentrations: 4.25 percent, 4.7 percent, 5 percent, and 5.5 percent. Values obtained for f_h and F_o were almost identical for all suspensions, indicating that a considerable variation in the concentration of bentonite, as used for conduction heating, had no significant effect on the rate of heat penetration.

Effect of Vacuum on the Heating Rate. Tests were made on one percent bentonite in No. 303 jars, with thermocouples located ¾ inch above bottom on the vertical axis of the jar. High vacuum jars were sealed with White caps under 25 inches of vacuum, and low vacuum jars were sealed with White caps under 5 inches of vacuum. Jars having no vacuum were sealed by means of clamps. The process temperature was 240° F., with come-up times of 12 to 13½ minutes.

The results of 5 runs, summarized in Table 1, indicate that vacuum has no significant effect on the heating rate of one percent bentonite in No. 303 jars. Later work showed, however, that vacuum has a pronounced effect on the cooling rate of five percent bentonite in the larger jars. Values obtained for jars of one percent bentonite sealed with no vacuum are similar to those shown in the table. Processes in Table 1 were calculated by the general method.

TABLE 1

Effect of Vacuum on Process Necessary to Obtain $F_o = 8$ One Percent Bentonite in No. 303 Jars

Vacuum	No. of Jars	Process in Minutes at 240° F. for $F_o = 8$		
		Minimum	Maximum	Average
5 inches	17	34.7	49.3	41.4
25 inches	15	35.7	49.4	40.8

Effect of Clamp vs. Side Entry on the Rate of Heat Penetration. Five runs were made to determine if clamps designed and built by the Owens-Illinois Glass Company had any effect on the rate of heat penetration when they were used to close jars. A comparison of the heating rate in jars closed with the clamp and having the thermocouple introduced through the top was made with jars sealed with White caps, and having the thermocouples introduced through the side. Two sizes of jars were used, Baby and No. 2½ filled with one percent bentonite. Jars sealed with White caps had a vacuum of 25 inches. All jars were processed caps-down.

The results, shown in Table 2, indicate no significant difference in the rate of heat penetration into one percent bentonite suspension between jars on which the clamp was used with cap entry of thermocouple, and jars sealed with the White caps, with side entry of thermocouple. Processes in Table 2 were calculated by the general method.

Size of Thermocouple Wire. Two runs were made at 240° F. and one at 250° F. to compare 24-gauge with 20-gauge thermocouple wire, in order to determine whether the greater thermal conductivity of the thicker wire would appreciably affect the temperature readings of couples in jars during the process. Tests were made

TABLE 2

Comparison of Clamp Technique and Side-Entry White Cap Technique
One Percent Bentonite

Size of Jar	Closure	Thermocouple	No. of Jars	Process in Minutes at 240° F. for $F_o = 8$		
				Minimum	Maximum	Average
Baby	Clamp	Through cap	6	30.3	31.5	30.9
Baby	White Cap	Through side	6	30.0	30.9	30.5
No. 2½	Clamp	Through cap	3	37.1	37.5	37.3
No. 2½	White Cap	Through side	3	36.4	37.0	36.6

with 1 percent, 3⅛ percent, and 5 percent bentonite suspensions in No. 303 and No. 2½ jars.

Results showed an appreciable increase in temperatures indicated by the thermocouples made with 20-gauge wire over those made with 24-gauge wire. As a result, all couples used in subsequent runs were made with 24-gauge wire.

Points of Slowest Heating. Forty-one runs were made to determine the points of slowest heating for one percent and five percent bentonite suspensions in Baby, Junior, No. 303, and No. 2½ jars, processed in three positions—caps up, caps down, and horizontal. The thermocouples were introduced in various ways, as shown in the photographs. For obtaining the desired data on jars processed in the horizontal position, three, and sometimes four, thermocouples were introduced into one jar.

The slowest heating points for the one percent suspensions were determined, in general, by inspection of the heat penetration data. However, when there was doubt as to the slowest heating position, F_o values were derived.

For the five percent suspensions, f_h and j values were determined, as well as the F_o value of the process and the cool. The lethality of the cool was ignored in deciding the points of slowest heating.

The results are summarized in Table 3. The points indicated are in reality the approximate centers of zones of slowest heating. A variation of ¼ inch on either side of the position indicated on the longitudinal axis appeared to make little difference in the results obtained.

Lethality of Cool. In connection with the processing of one percent bentonite, the possibility was considered of long cooling times adding more lethality to the process than short cooling times. Consequently, the F_o values for the cooling periods were evaluated in nearly all the preliminary runs, in which short and long cooling times were included (about 5 minutes to about 12 minutes).

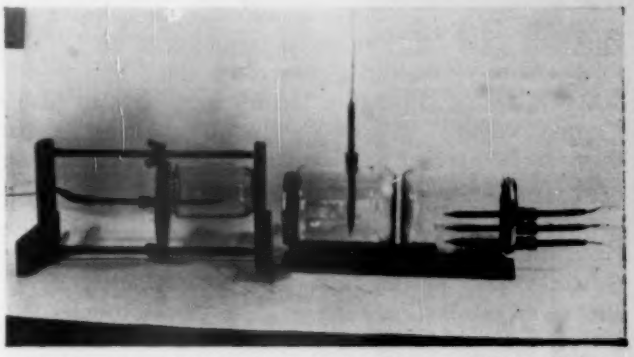

FIG. 3. Assemblies for Determining Slowest Heating Point.

No significant differences in the cooling rates were observed, except towards the end of the cooling cycle, when the lethality was little affected.

It was also anticipated that five percent bentonite would cool somewhat more slowly in jars than in cans of corresponding sizes. In most cases, however, the reverse was true, jars cooling much faster than cans. To determine if this was due to the greater headspace and higher vacuum in the jars, heat penetration measurements were made on cans having the same headspace (at least ½ inch), and high vacuum (above 20 inches). Under these conditions, rapid cooling also occurred in cans. The phenomenon must, therefore, be attributed to the boiling which takes place under reduced pressure in the containers, with consequent agitation of the contents and greatly increased rate of heat transfer. Under normal conditions, cans are filled full, and expansion of the contents during heating is taken care of by bulging of the ends. When the cans cool and sufficient vacuum is formed, the ends again become concave. Boiling of the contents, therefore, seldom occurs. In jars, however, the large headspace is maintained during the whole cooling period, with the result that boiling nearly always occurs when sufficient vacuum is formed.

General Investigations on Bentonite Suspensions

This phase of the investigation was carried out in order to obtain basic information on the comparative rate of heat penetration in jars and cans of various sizes. The objectives were:

1. To obtain fundamental data on the comparative heating rates of glass jars and cans.

2. To derive factors for converting an f_h value obtained on a product in a jar of one size to an f_h value for the same product in a jar of another size.

TABLE 3

Points of Slowest Heating
One Percent Bentonite

Jar	Size	Caps Up	Caps Down	Horizontal
Baby	200x309	½" above bottom	⅞" above cap	2¼" horizontally from cap and ⅛" above lowest extremity of jar
Junior	208x401	½" above bottom	⅞" above cap	2½" horizontally from cap and ½" above lowest extremity of jar
No. 303	303x411	½" above bottom	⅞" above cap	3¼" horizontally from cap and ⅜" above lowest extremity of jar
No. 2½	401x414	½" above bottom	⅞" above cap	3½" horizontally from cap and ¾" above lowest extremity of jar
		Five Percent Bentonite		
Baby	200x309	1⅛" above bottom	1⅜" above cap	1¼" horizontally from cap and 1⅛" above lowest extremity of jar
Junior	208x401	1½" above bottom	1⅞" above cap	2" horizontally from cap and 1⅜" above lowest extremity of jar
No. 303	303x411	2" above bottom	2½" above cap	2⅝" horizontally from cap and 1¾" above lowest extremity of jar
No. 2½	401x414	2" above bottom	2½" above cap	2⅝" horizontally from cap and 2¼" above lowest extremity of jar

3. To derive factors for converting an f_h value obtained on a product in a metal container to an f_h value for the same product in a glass container.

4. To derive correction factors for use with Ball's Formula Method of Process Calculations *(2)* so that they can be applied to the calculation of processes for glass containers heated and cooled under water.

Experimental Methods. As in the preliminary investigations, one percent bentonite was used in studies on convection heating and five percent bentonite in studies on conduction heating. The thermocouples and jars were assembled as described previously, the thermocouples being located at the respective slowest heating points. The jars and cans were placed upright in the retort, except for the study on effect of position.

When each retort load of containers was assembled, one process each was given at 212° F., 240° F., and 250° F. with a come-up time of approximately 5 minutes for both concentrations of bentonite, and one each at 240° F. and 250° F. with a come-up time of approximately 20 minutes in the case of one percent bentonite. In some cases, five percent bentonite was also processed with a come-up time of approximately 20 minutes at either 240° F. or 250° F.

Heat penetration determinations on at least three containers of each jar and can size were made for each processing cycle. Baby and Junior size jars and cans were processed together as were No. 303 and No. 2½ jars, and No. 2 and No. 2½ cans.

The cooling time for all runs was adjusted so that it took approximately 5 minutes for the temperature of the water in the retort to drop 100° F. At the end of

TABLE 4

Composite Heat Penetration Data on the Slowest Heating Containers Processed Upright in Water under Different Conditions

One Percent Bentonite							
No. of Containers Involved	*R.T.* (°F.)	*I.T.* (°F.)	*CUT* (min.)	*j*	f_h (min.)	f_h Range (min.)	Process[b] for F_0 or $F_{212} = 8$ (min.)
Baby Jars							
7	212	131	3.5	0.94	7.0	6.5- 7.0	13.8
10	240	130	5	1.00	6.9	5.8- 6.9	35.3
15	240	108	22	1.63	8.3	6.1- 8.3	30.1
11	250	123	6.75	0.80	6.5	5.3- 6.5	12.5
8	250	139	19	1.84	7.5	6.4- 7.5	11.2
Baby Cans							
9	212	130	3.5	0.61	4.1	3.1- 4.1	10.3
11	240	131	5	1.38	4.2	2.3- 4.2	31.2
9	240	133	20	1.44	3.5	2.0- 3.5	28.8
11	250	130	5	1.09	3.2	1.0- 3.2	9.3
9	250	138	19	4.36	4.4	2.4- 4.4	8.7
Junior Jars							
6	212	133	3.5	1.01	9.7	8.8- 9.7	17.6
10	240	128	5.25	0.89	8.8	7.7- 8.8	39.2
12	240	127	23	1.13	10.2	8.6-10.2	32.6
9	250	122	6.75	0.76	8.4	7.8- 8.4	14.1
6	250	136	19	1.20	9.3	8.4- 9.3	12.4
Junior Cans							
6	212	131	3.5	0.69	4.6	3.9- 4.6	11.4
9	240	130	5	0.87	4.4	2.6- 4.4	31.2
6	240	130	19.5	7.81	4.2	3.0- 4.2	29.4
8	250	129	5	0.82	3.5	2.0- 3.5	9.5
6	250	137	19	2.22	4.6	2.7- 4.6	8.3
No. 303 Jars							
6	212	131	9	1.21	11.4	10.1-11.4	15.5
33	240	135	8	0.73	12.0	9.2-12.0	40.5
19	240	114	24	0.75	14.6	9.7-14.6	36.2
13	250	129	6	0.87	10.7	8.4-10.7	18.3
7	250	130	21	1.15	12.8	9.3-12.8	16.4
No. 2 Cans							
6	212	131	8	0.52	6.3	5.4- 6.3	9.7
9	240	131	5	0.76	6.0	4.2- 6.0	33.1
8	240	124	23	0.64	10.6	6.0-10.6	31.8
9	250	126	5	0.71	5.0	4.2- 5.0	12.1
6	250	130	21	2.07	7.6	4.7- 7.6	11.1
No. 2½ Jars							
5	212	132	9	1.31	14.7	13.2-14.7	20.2
10	240	129	5	1.02	13.8	12.2-13.8	44.0
22	240	122	22	1.02	16.5	11.8-16.5	41.0
10	250	129	5	0.94	13.4	12.4-13.4	23.4
6	250	130	21	1.50	14.7	13.4-14.7	18.7
No. 2½ Cans							
6	212	132	9	1.47	7.4	5.9- 7.4	10.6
12	240	131	5	0.75	7.1	5.0- 7.1	34.4
6	240	127	21	1.35	8.1	6.0- 8.1	31.6
9	250	128	5	0.79	6.0	4.1- 6.0	12.9
6	250	130	21	2.37	7.5	5.1- 7.5	11.5

Five Percent Bentonite							
No. of Containers Involved	*R.T.* (°F.)	*I.T.* (°F.)	*CUT* (min.)	*j*	f_h (min.)	f_h Range (min.)	Process[b] for F_0 or $F_{212} = 8$ (min.)
Baby Jars							
9	212	150	3.5	1.68	21.9	19.8-21.9	30.0
11	240	150	4.5	1.48	22.9	20.6-22.9	53.8
18	250	148	4.5	1.46	22.4	20.4-22.4	33.4
11	250	147	19	1.75	21.9	19.8-21.9	27.0
Baby Cans							
9	212	148	3	1.44	21.7	19.6-21.7	28.1
9	240	150	4.5	1.47	22.3	20.7-22.3	52.4
14	250	149	4.5	1.35	22.5	20.8-22.5	33.4
5	250	147	19	1.42	22.2	20.0-22.2	26.6
Junior Jars							
9	212	145	3	1.75	31.1	27.8-31.1	37.9
13	240	150	4.5	1.60	31.8	28.6-31.8	60.6
20	250	152	5	1.63	32.2	27.9-32.2	41.0
14	250	141	18	1.33	34.3	27.6-34.3	39.8
Junior Cans							
8	212	147	3	1.46	27.7	26.7-27.7	33.1
8	240	150	4.5	1.58	28.4	27.6-28.4	57.5
18	250	148	4.5	1.53	28.8	24.6-28.8	39.8
5	250	148	19	1.74	27.8	27.4-27.8	33.1
No. 303 Jars							
6	212	140	4.5	1.23	50.0	46.2-50.0	53.2
9	240	136	8.0	1.76	50.3	47.3-50.3	93.0
11	250	145	5.5	1.65	51.7	46.5-51.7	70.3
73	250	139.5	19	1.43	57.1	45.4-57.1	70.4
No. 2 Cans							
6	212	143	4.5	1.50	57.3	52.7-57.3	62.3
7	240	107	4.5	1.78	57.0	53.8-57.0	107.1
9	250	145	5.25	1.40	56.8	51.6-56.8	69.0
No. 2½ Jars							
8	212	145	5	1.79	77.5	70.2-77.5	84.2
35	240	150	4.5	1.76	78.8	70.6-78.8	119.3
5	240	150	21	1.77	81.2	76.1-81.2	116.5
40	250	140	5.5	1.74	78.0	71.2-78.0	94.5
18	250	134	19	1.50	82.6	72.0-82.6	89.8
No. 2½ Cans							
8	212	146	5	1.68	76.5	71.8-76.5	71.0
37	240	149	4	1.74	73.5	61.3-73.5	116.4
4	240	151	21	1.67	75.3	73.9-75.3	115.3
23	250	145	5.25	1.69	74.8	67.7-74.8	87.1

[b] Lethal value of cool included.

the series of three runs with five percent bentonite or of the series of five runs with one percent bentonite, a new set of containers was assembled, using fresh bentonite suspensions and different thermocouples. At least two separate retorting operations were performed under each set of conditions, i.e., every run was repeated at least once.

The heat penetration data of the slowest heating container and of the container having the fastest cool under each set of conditions are tabulated in Tables 4 and 5, respectively. The sterilizing values were calculated by the general method *(3)*, using composite curves consisting of the heating curve of the slowest heating container and the cooling curve of the container with the most rapid cool. The j values were calculated, using an e value of 42 percent.

Significance of Jar Position. In the preliminary tests, data were obtained on the influence of jar position on the rate of heat penetration in jars containing bentonite suspensions. Baby and Junior jars containing one percent bentonite were processed together, as were No. 303 and No. 2½ jars. The experiments were repeated with five percent bentonite. Two runs were made in each case, one at 240° F. and one at 250° F. Thermocouples were placed at the pre-determined points of slowest heating. The processes for $F_o = 8$ were similar for jars processed in the horizontal position and for those processed with the caps down. Both of these positions permitted a slightly faster rate of heat penetration than did the cap-up position. For the five percent bentonite suspensions, no appreciable difference in the rate of heat penetration was observed with jars processed in any of the three positions.

In many of the experimental runs, heat penetration data were obtained with jars in both the cap-up and the cap-down positions. A summary of all the data obtained, where comparisons might be made, is presented in Table 6. The calculated processes for F_o or $F_{212} = 8$ are based on the slowest heating container in each group. The cool is included in the calculated processes, in order to present an overall picture of the effect of jar position on the heating rate. From these results, it may be seen that with one percent bentonite suspensions, the trend is for jars to heat a little more rapidly if they are in the cap-down position. However, these differences would be of little practical significance in terms of process times. As indicated previously, the jar position has little or no effect on the heating rate of five percent bentonite suspensions. Therefore, processes for products in glass containers need be determined only for the cap-up position.

Effect of the Length of the Come-Up Time on the Process. Data based on the slowest heating containers are presented in Table 7 to demonstrate the effect of come-up time on the length of the process. These data comprise the processes necessary to give an F_o of 8, calculated by the general method, for both a long and a short come-up time under the various conditions. The lethal value of the cool is excluded in these calculated processes in order to eliminate the effect of variations in the cool.

A come-up time of approximately 20 minutes had a significant effect in reducing the process requirements for cans and glass containers as compared with a come-up time of approximately 5 minutes. With one percent bentonite, the long come-up time shortened the process by from 0.3 to 6.3 minutes, while with five percent bentonite, the effect of the increased come-up time was greater, shortening the process by from 0.7 to 9.1 minutes. It was noted (Tables 4 and 8) that with one percent bentonite the long come-up time tended to produce heating curves having higher f_h and j values. This is attributed to the fact that the heating curve of a convection-heating medium tends to follow the retort heating curve more closely as the come-up time is increased. A result of this situation is that the true value of e for a come-up time, which is greater in length than the f_h value of the heating curve with nil come-up time, is less than it is with a short come-up time.

Comparison of the Rate of Heating of Jars and Cans in Water. Average heat penetration data and the processes necessary to produce an F_o or F_{212} of 8 in jars and cans are presented in Table 8. The containers were processed and cooled upright in water in the same retort. The data are averages of two to four separate processes, involving an aggregate of from five to ten different containers for each condition, except in the case of No. 2½ size containers processed at 240° F., where the results of only one run with five jars and four cans were available. The processes were calculated by the general method.

TABLE 5

Heat Penetration Data on the Fastest Cooling Jars Processed Upright in Water under Different Conditions

No. of Containers Involved	*R.T.* and *I.T.* of Cool (°F.)	Cool (min.)	Sterilizing Value of Cool (F_o or F_{212})
One Percent Bentonite			
Baby Jars			
5	212	4	0.51
25	240	4	0.18
19	250	5.25	0.75
Junior Jars			
9	212	3	0.58
23	240	5	0.15
15	250	6.75	0.62
No. 303 Jars			
6	212	6	0.76
50	240	5	0.26
20	250	4	0.96
No. 2½ Jars			
5	212	7	1.03
30	240	5	0.26
15	250	4	0.35
Five Percent Bentonite			
Baby Jars			
9	212	7	5.44
11	240	5.5	1.19
29	250	3.25	1.53
Junior Jars			
9	212	7	6.24
13	240	5.5	0.88
34	250	5	1.40
No. 303 Jars			
6	212	6	6.88
9	240	5	0.63
63	250	4.5	1.76
No. 2½ Jars			
8	212	6	6.40
35	240	5	1.76
42	250	4.5	2.30

Average data were used in this comparison of the rate of heating of jars and cans rather than the slowest data, because, if the latter were used, a comparison would be made, in most cases, between jars and cans processed at different times under different conditions. By using the average data, variations in the effect of retort operation (come-up time and cooling time) were minimized.

When filled with one percent bentonite suspensions (convection-heating), the jars heated more slowly than the cans of corresponding size when both were processed in water under the same conditions. For an F_o of 8, this difference amounted to approximately 3 to 10 minutes of process time, depending on the size of container. A long come-up time reduced this difference. For example, for a short come-up time (about 5 minutes), No. 2½ jars required 10.3 minutes longer than No. 2½ cans at 250° F., and 9.9 minutes longer at 240° F. For a long come-up time (about 20 minutes), the differences were 7.9 minutes at 250° F. and 7.3 minutes at 240° F.

TABLE 6

Significance of Jar Position on the Rate of Heat Penetration

Jar Size	No. of Jars	Position	R.T. (°F.)	I.T. (°F.)	CUT (min.)	j	f_h [1] (min.)	f_h Range (min.)	Process [1] for F_o or $F_{212} = 8$ (min.)
				One Percent Bentonite					
	7	Cap up	212	131	3.5	0.94	7.0	6.5- 7.0	13.8
	9	Cap down	212	130	3.5	0.98	7.3	6.1- 7.3	14.5
		Horizontal	....	....	....	....	....	....	....
	17	Cap up	240	130	19.5	2.63	7.6	5.8- 7.6	35.7
Baby	18	Cap down	240	132	20	2.45	7.2	5.0- 7.2	34.7
	2	Horizontal	240	129	6	1.14	5.8	5.7- 5.8	34.0
	20	Cap up	250	136	19	1.70	7.5	5.3- 7.5	10.7
	20	Cap down	250	139	19	2.00	7.4	5.1- 7.4	11.2
	2	Horizontal	250	131	8	0.91	5.5	5.5- 5.5	10.0
	6	Cap up	212	133	3.5	1.01	9.7	8.8- 9.7	17.8
	6	Cap down	212	134	3.5	0.91	9.0	7.9- 9.0	16.5
		Horizontal	....	....	....	....	....	....	....
	14	Cap up	240	132	20	2.11	9.0	7.7- 9.0	36.2
Junior	14	Cap down	240	131	19.5	1.93	8.2	6.9- 8.2	35.6
	2	Horizontal	240	129.5	6	1.12	7.3	7.0- 7.3	35.7
	14	Cap up	250	136	19	1.20	9.3	7.8- 9.3	12.4
	14	Cap down	250	136.5	19	1.29	8.4	6.8- 8.4	12.0
	2	Horizontal	250	131	8	1.10	7.2	6.7- 7.2	11.8
	6	Cap up	212	131	9	1.21	11.4	10.1-11.4	15.5
	6	Cap down	212	132	9	1.31	10.8	9.1-10.8	15.0
		Horizontal	....	....	....	....	....	....	....
	15	Cap up	240	128	21	0.92	12.4	9.2-12.4	35.3
No. 303	15	Cap down	240	128	21	1.46	11.3	8.8-11.3	33.1
	3	Horizontal	240	128	5	1.03	9.7	8.8- 9.7	37.4
	15	Cap up	250	130	21	1.15	12.8	8.4-12.8	16.4
	15	Cap down	250	130	21	1.67	10.4	8.2-10.4	14.5
	3	Horizontal	250	130	7.5	0.83	9.5	8.4- 9.5	16.7
	5	Cap up	212	132	9	1.31	14.7	13.2-14.7	22.3
	5	Cap down	212	132	9	1.16	13.3	10.5-13.3	18.3
		Horizontal	....	....	....	....	....	....	....
	14	Cap up	240	129	21	1.11	14.8	12.8-14.8	44.1
No. 2½	15	Cap down	240	129	21	1.02	14.0	11.1-14.0	42.7
	2	Horizontal	240	128	5	1.16	11.5	11.1-11.5	40.9
	14	Cap up	250	130	21	1.50	14.7	12.4-14.7	18.7
	14	Cap down	250	130	21	1.51	13.3	10.5-13.3	18.0
	3	Horizontal	250	130.5	7.5	0.83	11.3	10.0-11.3	18.2
				Five Percent Bentonite					
	2	Cap up	240	125	5.5	1.84	21.0	21.0-21.0	54.5
	2	Cap down	240	125	5.5	1.72	21.5	20.6-21.5	55.5
Baby	2	Horizontal	240	125	5.5	1.59	21.5	21.1-21.5	56.3
	2	Cap up	250	129	6	1.68	21.8	20.7-21.8	34.2
	2	Cap down	250	131	6	1.71	20.7	20.7-20.7	34.2
	2	Horizontal	250	130.5	6	1.53	21.6	21.3-21.6	34.2
	2	Cap up	240	124	5.5	1.81	30.7	29.9-30.7	72.0
	2	Cap down	240	124	5.5	1.72	31.5	29.8-31.5	72.0
Junior	2	Horizontal	240	123	5.5	1.71	31.9	31.1-31.9	72.3
	2	Cap up	250	131	6	0.89	30.6	30.5-30.6	46.5
	2	Cap down	250	131.5	6	0.88	31.5	30.8-31.5	46.7
	2	Horizontal	250	130	6	0.85	31.3	31.2-31.3	46.3
	3	Cap up	240	136	8	1.76	50.3	49.9-50.3	93.0
	3	Cap down	240	136	8	1.78	51.0	49.5-51.0	94.7
No. 303	2	Horizontal	240	136	8	1.76	51.8	51.2-51.8	95.0
	3	Cap up	250	145	5.5	1.65	51.7	50.1-51.7	70.3
	2	Cap down	250	145	5.5	1.67	53.0	50.4-53.0	68.0
	3	Horizontal	250	145	5.5	1.64	53.3	52.0-53.3	67.3
	3	Cap up	240	138	8	2.00	73.2	70.6-73.2	125.5
	3	Cap down	240	138	8	2.09	72.8	72.4-72.8	126.0
No. 2½	2	Horizontal	240	137	8	2.01	72.0	69.2-72.0	125.8
	3	Cap up	250	140	5.5	1.74	78.0	74.5-78.0	94.5
	2	Cap down	250	142	5.5	1.76	78.3	75.5-78.3	93.5
	2	Horizontal	250	141	5.5	1.71	77.5	74.0-77.5	94.0

[1] Includes cool.

[1] Value used in process calculations.

TABLE 7

Effect of Come-Up Period on Length of Process[k]

Size of Container	R.T. (°F.)	Jar				Can			
		No. of Tests	CUT (min.)	Process for $F_o = 8$ (min.)	Difference (min.)	No. of Tests	CUT (min.)	Process for $F_o = 8$ (min.)	Difference (min.)
One Percent Bentonite									
Baby	240	10	22	30.1	6.3	9	20	29.4	3.3
	240	10	5	36.4		11	5	32.7	
	250	8	19	11.9	1.6	9	19	9.3	1.1
	250	11	5	13.5		11	5	10.4	
Junior	240	13	23	33.8	3.9	6	19.5	30.1	1.7
	240	10	5.25	37.7		9	5	31.8	
	250	6	19	13.2	2.0	6	19	8.3	1.9
	250	9	6.75	15.2		8	5	10.2	
No. 303 Jar or No. 2 Can	240	19	24	38.2	3.3	8	23	32.8	0.3
	240	33	5.75	41.5		9	5	33.1	
	250	7	21	17.4	3.6	6	21	11.9	0.5
	250	13	6	21.0		9	5	12.4	
No. 2½	240	21	22	43.4	1.8	6	21	32.9	2.2
	240	10	5	45.2		12	5	35.1	
	250	6	21	19.9	4.1	6	21	11.9	1.9
	250	10	5	24.0		9	5	13.8	
Five Percent Bentonite									
Baby	250	23	19	31.8	5.4	5	19	30.5	5.8
	250	18	4.5	37.2		14	4.5	36.3	
Junior	250	22	18	41.6	6.9	5	19	39.8	6.1
	250	20	5	48.5		18	4.5	45.9	
No. 303 Jar or No. 2 Can	250	73	18	73.3	0.7				
	250	11	4.5	74.0					
No. 2½	240	5	21	134.0	3.4	4	21	123.2	6.2
	240	35	4.5	137.4		37	4	129.4	
	250	17	18	97.4	9.1				
	250	32	4.5	106.5					

[k] Process does not include lethality of cool.

Other sizes showed proportionate differences. In considering the processes at 212° F., it should be noted that there is a considerable difference in the "short" come-up time between the runs on Baby and Junior size containers and those on the No. 303 and No. 2½ sizes. For the larger sizes, this results in processes appreciably shorter than if a comparable come-up time had been used.

When heating occurred by conduction (five percent bentonite), Baby size jars and cans heated at about the same rate; however, differences were found in the larger containers. With the Junior size, the jars required a longer process than the cans to produce the same amount of lethality at the slowest heating point of the container; however, No. 303 jars heated at a more rapid rate than did the No. 2 cans. It must be remembered that, with the exception of the No. 2½ sizes, the diameters of the cans are greater than those of the corresponding jars (Baby can 202, jar 200; Junior can 211, jar 208; No. 2 can 307; No. 303 jar 303), which would have some influence on the comparative rates of heat penetration. In the case of the No. 2½ size containers, the average heat penetration data showed that the cans heated faster than the jars under some conditions, but under other conditions the reverse was true. The average f_h values of No. 2½ size jars and cans were similar, except in the case of the long come-up time at 240° F. However, in this case, the results are based on only one process involving four cans and five jars. These apparent inconsistencies in the comparative heating rates of the No. 2½ size containers are attributed to the use of average data. While the average f_h values for the jars and cans were quite similar, the j values for the jars were, in general, lower than those for corresponding cans.

Comparison of the Rate of Heating of Cans in Water and in Steam. The question arose as to whether cans heated with the jars in water under superimposed air pressure would have the same rate of heat penetration as when heated in the usual manner in steam. Identical processes were given cans of bentonite suspensions, in steam and in water at 250° F. and 240° F., leaving the containers undisturbed between runs. Because of the difference in the cooling procedure, depending on whether the processing medium was water or steam, the comparison of the sterilizing values of the processes in Table 9 is based on F_o values of the process alone without including the lethal value of the cooling period. These data represent the slowest heating cans in each process and each process involved at least three containers. The calculations of processes were made by the general method.

The results with one percent and five percent bentonite indicate that there is no significant difference in

TABLE 8

Comparison of Average Heat Penetration Data of Glass Containers and Cans Processed in the Same Retort Under Water

Size of Container	*R.T.* (°F.)	*CUT* (min.)	*j*		f_h		Process[1] for F_o or $F_{250} = 8$		
			Jar	Can	Jar (min.)	Can (min.)	Jar (min.)	Can (min.)	Difference (min.)
One Percent Bentonite									
Baby	250	6.5	1.04	1.66	6.0	1.8	12.2	7.5	4.7
Junior	250	6.75	0.99	1.02	8.1	2.5	14.7	7.7	7.0
No. 303 Jar or No. 2 Can	250	5	0.95	0.68	9.4	4.6	17.8	10.8	7.0
No. 2½	250	5	0.86	0.63	12.9	5.3	21.8	11.5	10.3
Baby	250	19	2.22	23.42[m]	7.1	3.2	11.0	7.5	3.5
Junior	250	19	1.57	11.83[m]	8.9	3.7	12.6	7.6	5.0
No. 303 Jar or No. 2 Can	250	21	1.55	2.79	11.1	6.2	15.1	10.2	4.9
No. 2½	250	21	1.37	2.68	13.9	6.8	18.4	10.5	7.9
Baby	240	5.25	0.94	0.64	6.0	2.8	33.9	29.1	4.6
Junior	240	5.25	0.97	0.59	8.3	3.1	36.4	29.7	6.7
No. 303 Jar or No. 2 Can	240	5.5	0.96	0.82	9.9	4.8	38.8	31.8	7.0
No. 2½	240	5.5	1.01	0.82	13.4	5.5	42.6	32.7	9.9
Baby	240	20	2.88	368.0[m]	6.9	2.5	31.6	27.8	3.8
Junior	240	19.75	2.06	24.3[m]	8.9	3.4	33.6	28.6	5.0
No. 303 Jar or No. 2 Can	240	21	1.29	2.15	11.2	6.7	34.6	31.2	3.4
No. 2½	240	21	1.28	2.00	14.2	7.2	38.9	31.6	7.3
Baby	212	3.5	1.04	0.72	6.7	3.6	14.3	10.2	4.1
Junior	212	3.5	1.06	0.69	9.3	4.2	17.0	10.8	6.2
No. 303 Jar or No. 2 Can	212	8.5	1.07	1.02	10.7	5.8	15.1	9.8	5.3
No. 2½	212	8.5	1.03	1.28	13.8	6.5	18.0	10.4	7.6
Five Percent Bentonite									
Baby	250	4.8	1.52	1.48	21.5	21.7	31.7	32.0	—0.3
Junior	250	4.8	1.46	1.57	29.8	28.3	40.3	38.7	1.6
No. 303 Jar or No. 2 Can	250	4.8	1.28	1.67	48.3	54.1	59.2	68.9	—9.7
No. 2½	250	4.8	1.33	1.74	72.9	72.3	84.7	86.4	—1.7
Baby	250	19	1.34	1.53	21.0	21.0	25.7	25.2	0.5
Junior	250	19	1.29	1.61	29.2	27.5	34.8	32.0	2.8
Baby	240	4.5	1.45	1.53	21.4	21.5	52.6	53.2	—0.6
Junior	240	4.4	1.47	1.62	30.3	28.0	62.3	56.7	5.6
No. 303 Jar or No. 2 Can	240	4.6	1.09	1.63	48.0	55.7	89.5	105.5	—16.0
No. 2½	240	4.2	1.35	1.75	74.1	74.1	124.0	123.8	0.2
No. 2½	240	21	1.74	1.71	78.8	75.7	116.7	114.6	2.1
Baby	212	3.2	1.61	1.64	20.8	20.8	28.0	28.5	—0.5
Junior	212	3.2	1.56	1.67	29.9	27.1	35.6	34.1	1.5
No. 303 Jar or No. 2 Can	212	6.5	1.18	1.68	48.4	54.9	49.1	59.8	—10.7
No. 2½	212	6.1	1.26	1.76	72.4	73.4	68.3	74.2	—5.9

[1] Includes lethal value of cool.
[m] Can temperature followed *R.T.* (long *CUT* and small container).

process requirements between cans heated in steam and cans heated in water, provided that the same length of come-up is used and the slower rate of cooling under water as compared to the usual cool by means of a spray is ignored. This is in confirmation of the findings of Bigelow, et al. *(3)*, who worked with starch suspensions and food products.

f_h Conversion Factors Derived from the Bentonite Studies. The f_h values of the slowest heating jars of bentonite processed under water and of cans of bentonite processed in steam were used to calculate the f_h conversion factors in Tables 10 and 11. By the use of the factors in Table 10, the f_h values of a convection-heating type product (one percent bentonite) or of a conduction-heating type product (five percent bentonite) can be calculated for jars of various sizes provided that the f_h value of one jar size is available. By use of the bentonite factors in Table 11, the f_h value of a jar processed in water can be derived from that of a can processed in steam or vice versa. It should be emphasized that these factors were derived from data obtained by using bentonite suspensions. It has not been proved that these factors hold true for actual food products.

Analysis of Heat Penetration Data on Glass Containers

The formula method of process calculation, as proposed by Ball *(1, 2)*, applies specifically to cans processed in steam and cooled by a water spray as in the common commercial procedure. This method assumes the same rate of cooling as of heating. These conditions are approximated in a steam process, but not in a water cook for glass containers because the large volume of hot water in the retort prevents rapid cooling. The lethal value of the come-up time, in terms of process time at retort temperature, for a steam process for cans was found by Ball *(1)* to approximate 42 percent of the come-up time, and this value is used in the mathematical calculation of processes for cans by the formula method. Whether or not this value was applicable to glass containers processed under water was not known.

In view of the above facts concerning the heating and cooling characteristics of glass containers processed in water as compared to cans processed in steam, it was necessary to determine the relationship between the assumed sterilizing value of the cooling time in the formula method and the value obtained with the shortest

TABLE 9

Comparison of Rates of Heating of Cans in Water and in Steam

Size of Container	Heating Medium	*R.T.* (°F.)	*I.T.* (°F.)	*CUT* (min.)	*j*	f_h (min.)	f_h Range (min.)	Process[a] for $F_0 = 8$ (min.)
One Percent Bentonite								
Baby	Steam	240	129	5	0.96	4.5	4.2- 4.5	33.3
	Water	240	131	5	1.38	4.2	- 4.2	32.7
	Steam	250	126	5	1.59	4.1	3.9- 4.1	12.7
	Water	250	130	5	1.09	3.2	3.0- 3.2	10.4
Junior	Steam	240	128	5	0.96	4.1	3.9- 4.1	31.9
	Water	240	130	5	0.87	4.4	3.8- 4.4	31.8
	Steam	250	126	5	1.77	3.3	3.1- 3.3	11.7
	Water	250	129	5	0.82	3.5	3.3- 3.5	10.2
No. 2	Steam	240	129	5	0.94	5.1	4.4- 5.1	33.1
	Water	240	131	5	0.76	6.0	5.9- 6.0	33.8
	Steam	250	127	5	1.46	4.3	4.2- 4.3	12.5
	Water	250	126	5	0.71	5.0	4.7- 5.0	12.4
No. 2½	Steam	240	129	5	0.91	5.8	5.6- 5.8	34.2
	Water	240	131	5	0.75	7.1	6.4- 7.1	35.1
	Steam	250	127	5	1.48	4.9	4.6- 4.9	13.9
	Water	250	128	5	0.79	6.0	5.5- 6.0	13.8
Five Percent Bentonite								
Baby	Steam	240	150	4	1.45	23.0	20.4-23.0	59.3
	Water	240	150	4.5	1.47	22.3	21.9-22.3	58.0
	Steam	250	147	4.75	1.49	21.0	20.0-21.0	35.0
	Water	250	149	4.5	1.35	22.5	20.8-22.5	36.3
Junior	Steam	240	150	4	1.57	29.4	27.0-29.4	68.7
	Water	240	150	4.5	1.58	28.4	28.2-28.4	66.7
	Steam	250	149	4.75	1.73	27.0	26.5-27.0	44.8
	Water	250	148	4.5	1.53	28.8	24.6-28.8	45.9
No. 2	Steam	240	113	5	1.55	56.0	52.7-56.0	114.8
	Water	240	112	4.5	1.78	57.0	53.8-57.0	117.9
	Steam	250	146	5.5	1.75	53.6	52.0-53.6	77.6
	Water	250	145	5.25	1.40	56.8	51.6-56.8	75.5
No. 2½	Steam	240	115	5	1.70	72.4	69.6-72.4	138.6
	Water	240	149	4	1.74	73.5	61.3-73.5	137.2
	Steam	250	149	5	1.70	74.7	69.2-74.7	99.6
	Water	250	145	5.25	1.69	74.8	68.5-74.8	100.3

[a] Process does not include lethality of cool.

TABLE 10

Factors for Converting the f_h of Bentonite Suspensions in One Size Jar to Another

Given Jar Size	Desired Jar Size			
	Baby	Junior	No. 303	No. 2½
One Percent Bentonite				
Baby	1.000	1.230	1.760	1.989
Junior	0.815	1.000	1.430	1.618
No. 303	0.568	0.699	1.000	1.130
No. 2½	0.504	0.619	0.885	1.000
Five Percent Bentonite				
Baby	1.000	1.498	2.495	3.608
Junior	0.669	1.000	1.670	2.411
No. 303	0.401	0.599	1.000	1.445
No. 2½	0.278	0.415	0.692	1.000

TABLE 11

Factors Derived from Bentonite Data for Converting f_h of Cans Processed in Steam to f_h of Glass Jars Processed in Water

Size of Container	Concentration	f_h		Factor f_h Jar/ f_h Can
		Jar	Can	
	Convection-heating			
Baby Can to Baby Jar	1% Bentonite	8.3	4.5	1.845
Junior Can to Junior Jar	1% Bentonite	10.2	4.1	2.485
No. 2 Can to No. 303 Jar	1% Bentonite	14.6	5.1	2.861
No. 2½ Can to No. 2½ Jar	1% Bentonite	16.5	5.8	2.842
	Conduction-heating			
Baby Can to Baby Jar	5% Bentonite	22.9	23.0	1.00
Junior Can to Junior Jar	5% Bentonite	34.3	29.4	1.17
No. 2 Can to No. 303 Jar	5% Bentonite	57.1	56.0	1.02
No. 2½ Can to No. 2½ Jar	5% Bentonite	82.6	72.4	1.14

practicable cooling time for glass. An evaluation of the come-up time for jars in water, analogous to the 42 percent figure applied to cans in steam, was needed also.

A procedure for calculating the lethal value of the come-up time and the cool correction was suggested by Ball as follows:

A. Come-up Time

1. Choose a heating curve representing a process in which there is an extreme lag in the rate of rise of temperature of the retort in the first part of the come-up time—the greatest lag that would ever occur. Determine the *j* value of this curve.

   NOTE: The *j* value of a heating curve which applies to a process in which the come-up time is nil is the correct *j* value for the heating curve of the same container in a process at the same temperature having a come-up time of any length which is not greater than the f_h value of the heating curve applying to nil come-up time.

   There was in this experiment, of course, no process having nil come-up time from which the correct *j* value for a heating curve could be taken.

   For all heating curves, when the come-up time was short, the value of *j* was assumed to be that which gave an *e* value for the curve of 42 percent. For products heating entirely by conduction, this *j* value was assumed to apply to the heating curve for the same container in all processes at the same temperature, regardless of the come-up time used.

   For heating curves applying to products heating entirely by convection, when the come-up time was long, just as with the short come-up time, the value of *j* was assumed to be that which gave an *e* value of 42 percent. This course was followed even when the length of the come-up time was greater than the f_h value of the heating curve, since the usual method of finding the value of *j* cannot be applied in the cases in which the value of f_h is changed by variation in the length of come-up time.

2. By the formula method, calculate the amount of lethal heat, expressed as percentage of the heat necessary to sterilize, at the center of the container, up to a given time, not including the effective lethal heat during cooling of the container. Do not make an adjustment for come-up time lethality effect. The time chosen should be in the neighborhood of that which would give sterilization. Designate this time by the symbol, *b*. This calculation method is given in problems VI-A and VI-B of "Mathematical Solutions . . . " *(2)*, where the time, *b*, is designated by the symbol, *x'*.

3. By the general method, using the same heating curve as was used in Step 2, determine the time required to put the same percentage of lethal heat into the center of the container, without including any part of the cooling curve. Designate this time, reckoned from the time the retort reaches holding temperature, by the symbol, *d*.

4. Let *c* represent the come-up time, and *e* represent the lethal value of the come-up time, in

terms of holding time, expressed as percentage of come-up time.

We have: $e = 100 \frac{b-d}{c}$ (1)

B. Cooling Time

1. Consider the heating and cooling curve of a jar in a process having a come-up time of M minutes. The curve should show the most rapid cooling it is possible to employ.
2. Let G_M be a process, determined by the general method,
   B_M be a process, determined by the formula method, adjusted by Procedure A for the lethality value of come-up time M,
   C_G be the sterilizing value of the cooling period in the process determined by the general method, expressed in terms of minutes holding time,
   C_B be the sterilizing value of the cooling period in the process determined by the formula method, expressed in terms of minutes of holding time. This value is automatically set by the calculation method.
3. Determine G_M and B_M for the process having come-up time, M, using the value, e (eq. 1), in making the come-up time adjustment in process B_M.
4. $B_M - G_M = C_G - C_B = C_{BG}$ (2)
   C_{BG} is the correction to be subtracted from B_M to adjust for the greater lethal value of the cooling period in the glass jar process as compared to that in the steam process in which cooling is effected as rapidly as possible, as assumed in the formula method.
5. For general application, the correction should be made as a percentage correction, represented by the formula:

$$\frac{100\ C_{BG}}{B_M} \quad (3)$$

In using the above mathematical method for calculating e values and cool corrections, an $m + g$ of 130° F. was employed, since this value more nearly represents the conditions in existence in processing and cooling jars under water at temperatures up to about 250° F. than does an $m + g$ of 180° F. For calculating the cool corrections, the actual e value, as determined for each particular curve, was used.

Calculations of Cool Correction and Lethal Value of the Come-Up Time. Composite heating and cooling curves were constructed from data on all sizes of glass containers (No. 2½, No. 303, Junior, and Baby), processed at 240° F. and 250° F. in the cap-up position (Tables 4 and 5). These curves represent the slowest heating rate, as determined by the f_h value, and the fastest cool, as determined by the actual F_o value of the cooling period, of the available data. That is, these curves represent a process having the lowest sterilizing value possible from the available data. Composite curves were constructed for short and long come-up times (approximately 5 minutes and 20 minutes). A fast cool (approximately 5 minutes) was used in all cases.

From these curves, the cool corrections and the lethal values of the come-up times were determined. The results of these calculations are presented in Table 12.

The minimum lethal value of the come-up time, in terms of holding time at retort temperature, amounted to 35 percent of the come-up time. The values ranged between 35.0 and 77.8 percent with a mean of 47.9 percent. From a consideration of the data, it was decided to accept for glass containers an e value of 42 percent, the same as is commonly employed in calculating processes for cans.

The cool correction factors varied considerably. In many cases, the glass jars were found to cool at a higher rate than that assumed for cans in the formula method of process calculations. The maximum cool corrections found for the different sizes of containers are shown in Table 12, and may be summarized as follows:

Jar Size	Cool Correction (Percent of calculated process time to be added to calculated process time)
Baby	1.3
Junior	3.1
No. 303	4.3
No. 2½	4.8

Investigations on Food Products

In order to check the validity of the results obtained in the bentonite studies, five food products were selected for investigation, namely, strained peas in Baby cans and jars; strained mixed vegetables with bacon in Baby cans and jars; chopped mixed vegetables with chicken in Junior cans and jars; peas in brine in No. 2 cans and No. 303 jars; and whole kernel corn in brine in No. 2 cans and No. 303 jars. Inoculated packs were made and heat penetration data were obtained on the above products packed under commercial conditions. Cans were processed in steam and jars in water. Spores of the putrefactive anaerobe, N. C. A. No. 3679, were used for inoculation, and thermal death time determinations were made on the suspension in the products at the same time the experimental packs were put up.

Heat Penetration Data. Heat penetration data, obtained on each product by the techniques used in the general runs on bentonite suspensions, are shown in Table 13. It is noted that there is relatively good agreement between the f_h conversion factors determined for bentonite and those determined experimentally from the three conduction-heating products investigated in the Baby and Junior jars and cans.

Experimental Packs. Approximately 12,000 spores of No. 3679 were inoculated into each container of strained and chopped foods, and 50,000 into each container of peas and corn. The strained and chopped foods were batch-inoculated before filling, so that the spores were evenly distributed throughout the product. The jars and cans of peas and corn were inoculated individually, just prior to sealing, by delivering one ml. of spore suspension into the center of the container.

For the Baby and Junior products, the process schedule included times from 45 minutes to 65 minutes

TABLE 12

Calculated c Value and Cool Correction from Composite Curves from Runs No. 1-134

No. of Curves		Bentonite (Percent)	*I.T.* (°F.)	*R.T.* (°F.)	*CUT* (min.)	Cool (min.)	*j*	f_h (min.)	General Method		Math Method	*c* (Percent)	Cool Correction (Percent)
Process	Cool								Exclu. Cool (min.)	Inclu. Cool (min.) GM	Inclu. Cool (min.) BM		
Baby Jars, Cap-up, Water Process													
10	25	1	130	240	5	4	0.91	6.9	36.4	35.8	35.3	38.0	—1.3
15	25	1	108	240	22	4	2.18	8.3	32.5	31.9	32.3	47.4	1.3
11	19	1	123	250	6.8	5.2	0.96	6.5	13.7	12.5	13.4	49.6	6.8
8	19	1	139	250	19	5.2	1.84	7.5	12.2	11.5	11.5	42.6	0.4
11	11	5	150	240	4	5.5	1.55	22.9	60	56.0	57.7.	57.5	3.0
18	29	5	148	250	4.5	3.2	1.52	22.4	37.4	34.9	35.2	51.1	0.8
11	29	5	147	250	19	3.2	1.52	21.9	32.3	30.1	29.5	39.0	—2.0
Junior Jars, Cap-up, Water Process													
10	22	1	128	240	5.2	5	0.89	8.8	38.9	38.4	37.3	43.8	—3.1
12	22	1	127	240	23	5	1.13	10.2	34.4	33.5	32.6	42.4	—2.6
9	15	1	122	250	6.8	5.5	0.84	8.4	16.3	15.2	15.3	48.9	0.6
6	15	1	136	250	19	5	1.44	9.3	13.7	13.0	13.0	42.6	0.0
13	15	5	150	240	4.5	5.5	1.63	31.8	73.9	69.0	71.3	45.0	3.2
20	36	5	152	250	5	4.5	1.63	32.4	50.0	48.3	47.0	46.1	—2.8
14	36	5	141	250	19	4	1.63	34.3	43.9	42.2	41.5	51.1	—1.7
No. 303 Jars, Cap-up, Water Process													
33	44	1	135	240	8	5	0.87	12.0	41.3	40.5	40.0	52.5	—1.1
19	44	1	114	240	24	5	0.79	14.6	38.9	37.9	36.3	43.4	—4.3
13	20	1	130	250	6	5	0.79	10.7	30.4	19.4	18.6	35.0	—4.1
6	20	1	130	250	21	5	1.17	12.8	17.6	16.6	16.6	42.5	—0.5
9	9	5	136	240	8	5	1.94	50.3	101.6	100.5	98.5	65.0	—2.0
11	62	5	145	250	5.5	4.5	1.65	51.7	77.1	71.8	70.2	38.2	—3.2
73	62	5	140	250	19	4.5	1.65	59.8	74.4	71.5	72.5	49.4	1.2
No. 2½ Jars, Cap-up, Water Process													
10	30	1	129	240	5	5	1.06	13.8	46.8	44.4	44.2	48.0	—3.8
22	30	1	122	240	22	5	0.92	16.5	43.8	42.9	40.9	39.1	—4.8
10	16	1	129	250	5	4.5	0.99	13.4	23.7	23.3	22.6	48.0	—3.2
6	16	1	130	250	21	4	1.35	14.7	19.6	19.3	18.6	44.7	—3.5
35	35	5	150	240	4.5	5	1.86	76.8	138.0	132.8	131.8	77.8	—0.8
5	35	5	150	240	21	7.5	1.86	81.2	134.5	129.7	128.1	50.0	—1.2
40	55	5	140	250	5.5	6	1.78	78.0	106.4	105.1	100.6	54.5	—4.2
18	55	5	134	250	19	4	1.78	82.6	99.1	96.0	99.2	56.3	3.2

TABLE 13

Heating Characteristics in Jars and Cans at a Retort Temperature of 240° F.

Product	Size of Container	*j*	f_h	f_2	x'_{bh}	f_h Conversion Factor jar/can	
						Food	Bentonite[a]
Strained peas	202x214 can	1.73	22.5	....	....		
	200x309 jar	1.84	23.2	....	....	1.03	1.0
Strained vegetables with bacon	202x214 can	1.52	23.9	....	....		
	200x309 jar	1.60	25.2	....	....	1.05	1.0
Chopped vegetables with chicken	211x210 can	1.69	29.7	....	....		
	208x401 jar	1.67	33.7	....	....	1.13	1.17
Peas (in brine)	307x409 can	0.866	5.9	13.5	7.4		
	303x411 jar	0.91	13.2	....	....		
Whole kernel corn (in brine)	307x409 can	0.38	13	19.8	12.7		
	303x411 jar	0.83	15	20.3	20.8		

[a] From Table 10.

at 240° F. at 5-minute intervals. Approximately 100 inoculated containers and 48 uninoculated controls were used for each process. For the brine packed peas, the process schedule included times from 25 minutes to 45 minutes at 240° F. at 5-minute intervals and for the whole kernel, brine packed corn, from 35 minutes to 55 minutes at 240° F. at 5-minute intervals. Forty-eight inoculated containers and 24 uninoculated controls were used for each process. The containers were incubated for a minimum of 4 months at approximately 30° C. (86° F.), and spoilage was determined by the formation of gas in the container. Doubtful containers were subcultured. No spoilage was encountered in any of the uninoculated controls.

A comparison of theoretical and derived processes is shown in Table 14. The theoretical processes were calculated by Ball's method with modifications developed from the bentonite studies, and were based on thermal death time data obtained with No. 3679 in the products tested.

Representative spoilage results are presented in Table 15. The calculated process times and lethal values in Tables 14 and 15 have been adjusted for the come-up times actually used. The higher incidence of spoilage in jars receiving the same process as cans of comparable size substantiates the slower heating characteristics obtained on jars.

TABLE 14

Comparison of Theoretical and Derived Processes

Product	Size of Container	*R.T.* (°F.)	*CUT* (min.)	Thermal Death Time Curve		Calculated Theoretical Process (min.)	Experimentally Derived Process	
				F	*z*		Min.	Lethality *(F)*
Strained peas	202x214 can	240	7	3.5	14.3	50.9	60[p]	5.2
	200x309 jar	240	6.5	3.5	14.3	52.5	60	4.8
Strained vegetables with bacon	202x214 can	240	7	5.4	17.3	51.6	50	5.0
	200x309 jar	241	6.5	5.4	17.3	52.3	60	7.4
Chopped vegetables with chicken	211x210 can	240	6.5	3.75	15.6	58.9	65	5.0
	208x401 jar	240	6.5	3.75	15.6	65.3	65	3.7
Peas (in brine)	307x409 can	240	1	3.5	14.3	26.8	30	4.3
	303x411 jar	240	6	3.5	14.3	33.7	35	3.8
Whole kernel corn (in brine)	307x409 can	240	2	6.8	18	35.2	60[q]	13.7
	303x411 jar	240	15	6.8	18	40.0	45	8.1

[p] 1% spoilage at 60 minutes.
[q] 12.5% spoilage at 45 minutes; no spoilage at 50 minutes; 2% spoilage at 55 minutes.

Discussion. Heating characteristics of products having a straight line semi-logarithmic heating rate show good agreement with those predicted from bentonite studies and can data. It is noted that brine packed peas, which showed a broken rate of heating in No. 2 cans, showed a straight line semi-logarithmic rate in No. 303 jars. In general, products in jars appear to heat more slowly than the same products in cans. This difference is more pronounced in products heating by convection than in products heating by conduction.

Summary

Comparative heat penetration tests have been made on bentonite suspensions in glass and metal containers. One percent suspensions were used for studying convection-heating rates and five percent for conduction-heating rates. The use of bentonite of intermediate concentrations for broken heating curves did not prove feasible. Heating and cooling rates were determined in Baby, Junior, No. 303, and No. 2½ jars; and in Baby, Junior, No. 2, and No. 2½ cans.

Preliminary runs were made to establish the stability of the bentonite suspensions on repeated heating; the effect of vacuum on the heating and cooling rates; the best method of introducing thermocouples into jars; and the positions of slowest heating in jars processed cans-up, caps-down, and horizontally.

In the general runs, the following factors were studied:

1. Effect of a long as compared to a short come-up time. A long come-up time had a significant effect in reducing the process time required after the retort reached holding temperature.

2. Comparison of heating rates of jars and cans in water. With one percent bentonite, jars heated appreciably slower than cans of corresponding sizes. This effect was not so pronounced with the five percent bentonite, particularly in the smaller sizes of containers.

3. Comparison of heating rates of cans in water and in steam. Results showed no significant differences with either one percent or five percent bentonite.

4. Significance of jar position. With 1 percent bentonite, the cap-up position gave somewhat slower heating rates than either the cap-down or horizontal positions, which were about equal. Position appeared to have no appreciable effect on the heating rate of five percent bentonite.

5. Application of Ball's calculation method to processes for glass. It was found that Ball's method can be applied if the value of j is calculated on the basis of $c = 42$ percent of the come-up time and the proper cool correction is used.

Factors were calculated for converting the f_h of bentonite suspensions from one size jar to another, and for converting the f_h of cans processed in steam to the f_h of jars processed in water.

Inoculated experimental packs were made of five food products in glass and tin, and comparative heat penetration data obtained. Results approximated closely those obtained in process calculations based on heat penetration results with bentonite.

TABLE 15

Illustrative Spoilage Results from Inoculated Packs

Product	Size of Container	Process		Percent Spoilage	Lethality *(F)*
		Min.	*R.T.* (°F.)		
Strained peas	202x214 can	55	240	4	4.3
	200x309 jar	55	240	25	3.9
Strained vegetables with bacon	202x214 can	45	240	14	3.8
	200x309 jar	45	241	21	3.6
Chopped vegetables with chicken	211x210 can	60	240	5	4.0
	208x401 jar	60	240	14	2.9
Peas (in brine)	307x409 can	30	240	0	4.3
	303x411 jar	30	240	2	2.9
Whole kernel corn (in brine)	307x409 can	40	240	2	8.0
	303x411 jar	40	240	4	6.9

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